

#### TECHNICAL REPORT

WVT-6915

ON THERMAL CONDUCTIVITY OF COMPOSITES

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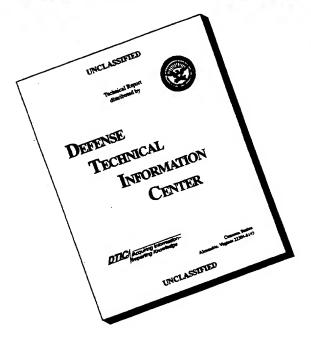
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#### WVT-6915

#### ON THERMAL CONDUCTIVITY OF COMPOSITES

TECHNICAL REPORT

BY

GIULIANO D'ANDREA

Doctoral thesis presented to and accepted by Rensselaer Polytechnic Institute, May 1969.

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#### ON THERMAL CONDUCTIVITY OF COMPOSITES

Abstract

 Thermal conductivity of composites, made of highly conductive metal fibres randomly distributed in low conductive matrices, is investigated experimentally as well as theoretically.

Experimental results are presented for a factorial experiment consisting of five factors at two levels. The factors are the length of the fibre, the cross section of the fibre, the thermal conductivity of the fibre, the thermal conductivity of the matrix, and the fibre volume ratio.

It is shown that the most meaningful factors to be considered for an optimum design of the types of composites in question are, in the order of importance, the conductivity of the matrix, the fibre volume ratio, and the fibre cross section.

Earlier theoretical efforts consisted of mathematical models which are highly idealized, e.g. square lattices, face centered cubic models (Maxwell, Rayleigh, Bruggeman). The model used in this investigation is that of a matrix filled with fibres, ellipsoidal in shape and randomly distributed, according to prescribed fibre volume ratios. It is a stochastic model. An expression of the conductivity of this composite is derived and results are obtained with the aid of a high-speed computer. Theoretical prediction of the stochastic model checks with experimental data. Also it shows that additional factors are involved in describing composites which are not covered by earlier theories. These factors are fibre geometry and its distribution.

Moreover, a specific specimen formulation gives a thermal conductivity similar to that of stainless steels but a density only a quarter of that of the steel. Cross-Reference Data

Composite Materials

Thermal Conductivity

Thermal Properties

Heat Transfer

Fibers (Metallic)

Epoxy Plastics

Manufacturing Methods

Test Methods

Statistical Analysis

PART I

#### INTRODUCTION

#### A. Historical Review

#### 1. Theoretical

Published work to date has cast light only on certain theoretical and experimental aspects of the thermal problems created by the presence of several phases of aggregates in an elastic medium. The object of this study is to describe experiments which serve to elucidate further the theory of composite thermal problems and, in particular, to show the significance of fiber geometry when metallic fibers are uniformly distributed in low thermal conductive matrices such as epoxies. The fundamental equation of the thermal conductivity, and the basic relation which serves as a definition of that term was proposed by Fourier in 1822:

$$Q = K A \frac{dT}{dX}$$
 (1)

where Q is the amount of heat flowing per unit time through an area A, and the temperature gradient in a direction perpendicular to A is  $\frac{dT}{dX}$ . The law states that the amount of heat flowing per second per unit area is proportional to the temperature gradient, and the proportionality constant K is the thermal conductivity.

Equation (1) is well established for conduction in homogeneous, isotropic solids and is also applied for a binary mixture with two phases in alternate layers or laminae. In this mixture, neither phase is continuous

in one dimension, and both phases are uniformly continuous in the other two directions. If the heat current is parallel to the laminae:

$$K_B = V_R K_f + (1 - V_R) K_m$$
 (2)

If the heat current is perpendicular to the laminae:

$$K_{B} = \frac{K_{f} K_{m}}{V_{R} K_{m} + (1 - V_{R}) K_{f}}$$
(3)

where  $K_B$  is the effective thermal conductivity of the binary mixture;  $V_R$ , the fiber volume ratio;  $K_f$ , the conductivity of the filament; and  $K_m$ , the conductivity of the matrix.

In 1892, Rayleigh<sup>2</sup> and Maxwell<sup>3</sup> proposed the dilute dispersion equation from which many of the relationships of composite thermal conductivity are derived. The equation

$$\frac{K_B - K_C}{K_R - 2K_C} = \left[V_R\right]_d \frac{K_d - K_C}{K_d - 2K_C} \tag{4}$$

is the basic equation obtained from references 2 and 3; subscript d denotes the discontinuous phase; and C, the continuous phase. Equation (4) is found in many forms.

In 1935, Bruggeman<sup>4</sup> developed the model of a cubical array of uniform, solid spheres in a continuous medium where no limit is set on the amount of the phases, and no phase is necessarily completely surrounded by another. The equation obtained was

$$V_{R_{1}} \frac{K_{1} - K_{B}}{K_{1} + 2K_{B}} + V_{R_{2}} \frac{K_{2} - K_{B}}{K_{2} - 2K_{B}} = 0$$
 (5)

where the subscripts 1 and 2 refer to phase 1 and 2 respectively. Bruggeman also derived, from the Rayleigh-Maxwell equation, another relationship applicable for dispersions of any concentrations<sup>4</sup>. Here the particles of conductivity  $K_1$  are dispersed in a continuous medium of conductivity  $K_2$ :

$$(1-V_R) = \frac{K_1 - K_B}{K_1 - K_2} \left[\frac{K_2}{K_B}\right]^{\frac{1}{3}}$$
(6)

In the same work, Bruggeman also derived an equation for a mixture with cylindrical shapes parallel to each other and with heat current perpendicular to the axis of the cylinders for a wide range of concentrations:

$$(V_R)_1 \frac{K_1 - K_B}{K_1 + K_B} + (V_R)_2 \frac{K_2 - K_B}{K_2 - K_B} = 0$$
 (7)

Equations (1) through (7) were then generalized for any shape of the phases as follows<sup>5</sup>:

$$(V_R)_1 = \frac{K_1 - K_B}{K_1 + FK_B} + (V_R)_2 = \frac{K_2 - K_B}{K_2 + FK_B} = 0$$
 (8)

where the various values of the factor, F, result in the previously discussed mixture equations for the different thermal models; i.e.,

F = 0 gives equation (3)

F = 1 gives equation (7)

F = 2 gives equation (5)

 $F = \infty$  gives equation (2)

Above models are based on exact mathematical calculations, but, in practice, one seldom encounters such simple geometries for particles of varying irregular sizes.

From 1935 and on, several intermediate formulae have been proposed either empirically or on the basis of some simple models.

In 1940 and 1941, NeKrasov<sup>6</sup> and Bogomolov<sup>7</sup> obtained expressions for heat conductivity in two extreme types of packing particles (grains):

a. For the cubic packing:

$$K_{B} = \frac{3}{2} \pi K_{P} \frac{0.9 - (V_{R})_{m}}{\left[2.1 + (V_{R})_{m}\right]^{2}}$$
(9)

b. For the tetrahedral packing:

$$K_B = 3 \pi K_a l_n \frac{43 + 0.31P}{P - 26}$$
 (10)

where

 $\boldsymbol{K}_{\boldsymbol{R}}$  is the thermal conductivity of the particles

 $\left( \text{V}_{R} \right)_{m}$  is the volume ratio of the matrix

K<sub>a</sub> is the thermal conductivity of the air gaps

P is the percentage of particle space (or porosity)

In 1961, Sugawara and Yoshizawa $^8$  developed an empirical relation between experimental values of  $K_B$  and volume ratio of matrix (or porosity)  $(V_R)_m$ ; i.e.,

$$K_{B} = (I - A) K_{P} + A K_{m}$$

$$(11)$$

where

$$A = \frac{2^{n}}{2^{n} - 1} \left\{ 1 - \frac{1}{\left[ 1 + (V_{R})_{m} \right]^{n}} \right\}$$

and n is an empirical exponent which they calculated as 6.5 for their sample.

In 1963, Brailsford and Major<sup>9</sup> summarized the expressions for the thermal conductivity of two phase media for various types of structures, by assuming that each particle of phase 1 is surrounded by a region of material of phase 2, which is in turn surrounded by material having an average conductivity equal to that which has to be calculated (Figure 1).

For the conductivity of a random distribution of solid spheres in a continuous medium, they found (by assuming that the temperature distribution in each region satisfies Laplace's equation  $\nabla^2 T = 0$ ).

$$\frac{K_{B}}{K_{2}} = \left( 1 - 2V_{1} \frac{1 - K_{1} / K_{2}}{2 + K_{1} / K_{2}} \right) / \left( 1 + V_{1} \frac{1 - K_{1} / K_{2}}{2 + K_{1} / K_{2}} \right)$$
(12)

where  $\mathbf{V}_1$  is the volume fraction of phase 1.

They also extended this model to determine the conductivity of a system containing three or more different phases, for example, a mixture in which spherical particles of material 1 or material 2 are embedded in material 0. The thermal conductivity of this system is given by:

$$K_{B} = \frac{\left\{ K_{0} V_{0} + K_{1} V_{1} \frac{3K_{0}}{(2K_{0} + K_{1})} + K_{2} V_{2} \frac{3K_{0}}{(2K_{0} + K_{2})} \right\}}{\left\{ V_{0} + V_{1} \frac{3K_{0}}{(2K_{0} + K_{1})} + V_{2} \frac{3K_{0}}{(2K_{0} + K_{2})} \right\}}$$
(13)

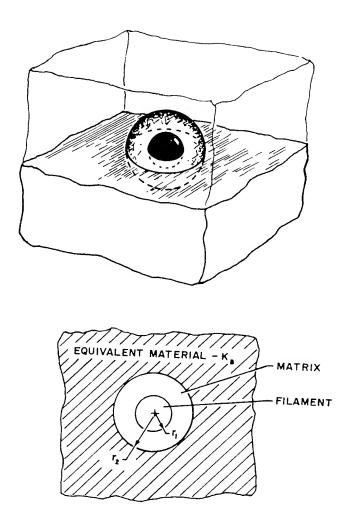


Figure 1. Thermal Model Used by Brailsford and Major.

Equation (13) assumes that phase "0" is continuous.

From (13) they also deduced, by letting  $K_0 = K_B$ , the conductivity for a random two phase containing regions of both single phases in the correct proportions, embedded in a random mixture of the same two phases, having a conductivity equal to the average value of the conductivity of the two phase assembly which is being calculated, i.e.,

$$K_{B}^{2} \left[ 6(-V_{1} - V_{2}) \right] + K_{B} \left[ 3V_{1} (2K_{1} - K_{2}) + 3V_{2} (2K_{2} - K_{1}) \right]$$

$$+ K_{1} K_{2} \left[ 3(V_{2} + V_{1}) + 1 \right] = 0$$
(14)

where  $V_2 = 1 - V_1$ .

In 1967, Springer and Tsai<sup>10</sup> introduced a thermal model which takes into consideration the shape of the filaments and their geometrical arrangement. The model is shown in Figure 2. The basic assumptions they made are:

- a. Constant temperature difference between  $X = \pm a$
- b. The total heat flow per unit length along the filament, may be divided in three independent parts.
- c. The temperature distribution at any local point in the element satisfies  $\nabla^2 T$  = 0.
  - d. The boundary conditions are:

at y = 0, y = 
$$\pm$$
 b, K  $\frac{\partial T}{\partial y}$  = 0

at x = a, T = 
$$T_a$$
, at x = -a, T =  $T_{-a}$   
at x = 0, T =  $\frac{\Delta T}{2}$  = constant

#### ARRANGEMENT OF FILAMENTS

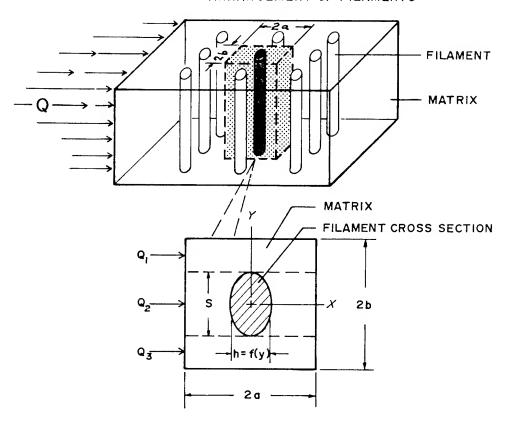


Figure 2. Thermal Model Used by Springer and Tsai.

e. The condition existing at the interface between the filament and matrix must be specified. For zero thermal resistance at the interface,

$$\begin{aligned}
T_f &= T_m \\
K_f \frac{\partial T}{\partial n} \Big|_f &= K_m \frac{\partial T}{\partial n} \Big|_m
\end{aligned}$$

where n is the direction normal to the interface.

With the above assumptions, the problem is then solved by imposing a known uniform temperature distribution ( $\Delta T$ ) between  $x = \pm a$  surfaces. For symmetrical packing arrays, the following general expression was obtained:

$$\frac{K_{B}}{K_{m}} \cong \left(I - \frac{S}{2b}\right) + \frac{a}{b} \int_{0}^{s} \frac{dy}{(2a-h) + (h K_{m}/K_{f})}$$
(15)

Note that for a square filament (S = X = constant), a = b and

$$\frac{K_{B}}{K_{m}} = 1 - \sqrt{V_{f}} + \frac{1}{\sqrt{1/V_{f} + B/2}}$$
 (16)

for a cylindrical filament (S = d), a = b, hence

$$\frac{K_{B}}{K_{m}} = 1 - 2\sqrt{V_{f}/\pi} + \frac{1}{B} \left\{ \pi - \frac{4}{\sqrt{1 - (B^{2} V_{f}/\pi)}} + \frac{1}{B^{2} V_{f}/\pi} \right\}$$

$$tan^{-1} \frac{\sqrt{1 - (B^{2} V_{f}/\pi)}}{1 + \sqrt{B^{2} V_{f}/\pi}}$$

$$(17)$$

where

$$B \equiv 2 \quad \left(\frac{K_m}{K_f} - I\right)$$

In 1968, Behrens<sup>11</sup> obtained thermal conductivity expressions by using the method of analysis introduced by Max Born<sup>12</sup> into solid state physics, where it is commonly known as the "method of long waves". It consists in considering plane waves, in this case thermal waves, traveling through the material with wavelengths which are long as compared with the inter-component spacings. By calculating their damping coefficients in the principal directions of the medium, explicit expressions for the average thermal conductivities can be obtained.

For the composite with a rectangular lattice having two constituents (and cubic particles), in the limit, Behrens gives:

$$\frac{K_{B}}{K_{m}} = \frac{(P+I) + (P-I) V_{R}}{(P+I) - (P-I) V_{R}}$$
(18)

For the composite with cubic symmetry (with spherical particles or inclusions), in the limit, Behrens gives:

$$\frac{K_{B}}{K_{m}} = \frac{(P+2) + (P-1) 2V_{R}}{(P+2) - (P-1) V_{R}}$$
(19)

where  $P = K_f/K_m$ .

In 1968, Hashin<sup>13</sup> generalized the "Self-Consistent Scheme" or SCS method of approximation for analysis of effective properties of particulate composites. The basic underlying assumption is that a typical basic element of a heterogeneous medium, such as a single crystal in a polycrystal or an inclusion in a particulate composite, can be regarded as being embedded in an equivalent, homogeneous medium whose properties are

the unknowns to be calculated. This model is the same as the one introduced by Brailsford and Major in 1963. The general equation obtained by Hashin

is 
$$2[2+c^{1}+\beta(1-c^{1})] \kappa^{2}-[2(1+2c^{1})+\beta(1-4c^{1})+$$
  
 $9(\beta-1) V_{2}] \kappa-[2(1-c^{1})+\beta(1+2c^{1})]=0$ 
(20)

where 
$$K = \frac{K_B}{K_I}$$
,  $\beta = \frac{K_2}{K_I}$ ,  $C^1 = \frac{q^3}{\rho^3}$ 

It is noted that if C' = 1, equation (20) reduces to equation (5), or

$$V_{1} \frac{K_{1} - K_{B}}{K_{1} + 2K_{B}} + V_{2} \frac{K_{2} - K_{B}}{K_{2} + 2K_{B}} = 0$$
 (21)

If  $C' = V_2$ , then

$$K_{B} = K_{I} \left[ 1 + \frac{V_{2}}{K_{1} - K_{1}} + \frac{V_{I}}{3} \right]$$
 (22)

This equation is also a rigorous result for a very special geometry, which is obtained when a volume is filled out ad infinitum with composite spheres of variable sizes. In each composite sphere, the volume ratio of the inner spherical core to matrix shell is  $V_2$ , and the conductivities of core and shell are  $K_2$  and  $K_1$  respectively.

Hashin, in his paper, introduced C' which he believed to be a new geometrical parameter "whose value is not easily assigned."

NOTE: If a closer look is taken at  $C' = \frac{a^3}{\rho^3}$ , it is found that C' is none other than the volume ratio of the particle to the matrix, and its value has a range of 0 to 100 percent.

#### 2. Experimental

In 1789, Inger-Hausz<sup>14</sup> developed one of the first techniques for measuring conductivities. The basic apparatus was of a comparative type; identical bars of different materials were coated with wax, and one end of the bars was heated at a constant temperature. When a steady state was reached, the wax was melted to different distances from the source on the various rods. By observing these distances, the relative thermal conductivities could be determined by the equation:

$$\frac{\mathsf{K_1}}{\mathsf{K_2}} = \frac{\mathsf{X_1}^2}{\mathsf{X_2}^2} \tag{23}$$

If the bars are of great length, the assumption of infinite length that is necessary for this equation is nearly satisfied.

In 1861, Angstrom<sup>15</sup> used the impulse method of measurement of thermal conductivity. This method actually yields the thermal diffusivity,  $\alpha = \frac{K}{\rho C}$ , so that it is necessary to have an accurate knowledge of specific heat and density as a function of temperature in order to determine the conductivity. The basic apparatus is a long bar or wire with an electric heater at one end. The other end extends into homogeneous surroundings of air or an insulating medium. A steady state temperature gradient

is imposed on the specimen and a heat pulse varying sinusoidally with time is superimposed on the hotter end. This temperature disturbance is propagated down the bar at a rate that varies with the thermal diffusivity.

In 1867, Forbes<sup>16</sup> introduced the "Forbes Bar Method". He used a wrought iron bar one inch in diameter and eight feet long, one end of which was in a bath of molten lead. The other end extended into still air. Heat was lost from the surface of the bar by convection and radiation. This introduced a longitudinal temperature gradient into the bar, and when a steady state was reached, this gradient was measured. The basic equation used was:

$$Q = KA \frac{dT}{dX}$$
 (24)

or, differentiating and rearranging:

$$K = \frac{1}{A} \frac{dQ}{dX} \frac{1}{d^2 T / dX^2}$$
 (25)

Following this experiment, the entire bar was heated to a high temperature and allowed to cool while measurements of temperature were being made as a function of time. In the cooling part of the experiment, the heat radiated comes completely from the heat content of the bar. An accurate knowledge of specific heat and density is necessary to find this thermal energy. The energy lost per second from the bar per unit length is:

$$\frac{dQ}{dX} = CA\rho \frac{dT}{dt}$$
 (26)

In 1900, Kohlrausch <sup>17</sup> devised an electrical method for determining conductivity. He heated a rod by passing an electric current through it while holding the ends at the temperature of the surrounding. The method yields the ratio of thermal and electrical conductivities. The latter may be determined independently and accurately, therefore, the thermal conductivity is readily calculated. The necessary measurements include (1) the potential drop between two points equally distant from the midpoint, hence at the same temperature, (2) the temperature at these points, and (3) the temperature, which is a maximum, at the middle of the bar. The equation used was

$$\frac{K}{\sigma} = \frac{(dV/dX^2)}{J(d^2T/dX^2)}$$
 (27)

where

 $\sigma$  is the electric conductivity (1/ohm)

V is the electric potential (volts)

J is the conversion factor

In 1935, Fitch 18 used a straightforward method of measuring conductivity by maintaining a plate of material with one surface at a given temperature, then measuring the temperature of the opposite surface when a steady state of heat flow was obtained. His apparatus was unique in the use of a copper block for a calorimeter. This apparatus is quite satisfactory for low conductivity materials, but for metals which have high heat conductivities, only a small temperature drop is encountered across the sample, leading to inaccurate conclusions. When one has a simple, pressed joint between two materials, as between the heat source and the specimen, a large and variable temperature drop is noted. This

drop often accounts for a large part of the temperature difference that is measured in connection with such a determination. This method is, thus, not particularly well suited for metals.

In 1952, The American Society for Testing Materials 19 prescribed a method (Specs. C177-45) in which two plates of the material under test are placed on opposite sides of a heater. Measurements of the steady state temperatures of both sides of each plate gives data for the calculation of the conductivity. The assumption is made that all of the heat generated passes through these plates, and if a suitable guard heater is supplied around the edges, this condition is approximately achieved.

In the same year (1952), Mikol<sup>20</sup> modified the parallel plate method by using a hollow cylinder of the test material and by heating this cylinder at the center. The heater is in the axial hole and suitable guard heaters are provided at the ends. The quantity of heat flowing through the material is known precisely with the experimental arrangement. The conductivity is determined from the relationship.

$$K = \frac{Q \ln (r_2 / r_1)}{2(T_1 - T_2)}$$
 (28)

In a commonly used experimental arrangement, the exterior of the cylinder is cooled by radiation either into the atmosphere or into a region of elevated temperature. This method is called the Radial Heat Flow Method.

In combatting "end heat-losses," Adams and Loeb<sup>21</sup>, in 1954, and others have used a spheroidal shaped specimen that completely surrounds an electric heater. This method is quite well suited for the investigation of the conductivity of ceramic and insulating materials.

In 1964, Goldsmid<sup>22</sup> summarized the experimental methods and results of measurements on solids and fluids, as well as theories of heat conductions. From his discussion, it is evident that the modern equipment for measuring thermal conductivity is still of the same type discussed earlier, most of the work is devoted to improve the methods of measurements by making them simpler, extending their range of applicability and, above all, by increasing their accuracy.

The two apparati used in this study are the thermoconductometer and a comparative thermal conductivity measuring system which are described in detail in Part III.

#### B. Statement of the Problem

No thermal data on composite materials made of high conductive metal fibers uniformly distributed in low conductive matrices is presently available in the literature.

The property of interest in this study is the average thermal conductivity as it varies in relation with the following parameters: length of the fiber, cross section of the fiber, thermal conductivity of the fiber, thermal conductivity of the matrix, and fiber volume ratio.

In addition to providing reliable thermal conductivity data, it is desirable to construct a theoretical model which correlates the average thermal conductivity and the aforementioned physical parameters.

#### PART II

#### **THEORY**

#### A. Thermal Conductivity of Composite Materials

The following review is necessary to develop a stochastic thermal conductivity model. Since the model is controlled by probabilistic laws, this study investigates a collection of random variables from the point of view of their interdependence and limiting behavior.

#### 1. Probability of an Event

Let

x, y,  $x_i$ ,  $y_k$ ,  $z_{ik}$  = outcomes of a random experiment S = the set of all outcomes = outcome space  $\beta$  = a family of certain subsets of S = events

If  $\beta$  satisfies the following axioms:

(D) The outcome space and the enpty set are in eta :  $s \in eta$  ,  $\theta \in eta$ 

(B2) If each set of the finite or countable sequence A1, A2, ... A5 ... is in  $\beta$  , then their union and intersection are in  $\beta$  :

Ai 
$$\in \beta$$
 for  $i = 1, 2, \dots, \bigcup_{(i)} Ai \in \beta$   
Ai  $\in \beta$  for  $i = 1, 2, \dots, \bigcap_{(i)} Ai \in \beta$ 

(B<sub>3</sub>) If a set  $\Lambda$  is in  $\beta$ , then its complement is in  $\beta$ :  $A \in \beta \rightarrow \overline{A} = S - A \in \beta$ 

then, eta is called a Borel field on S. Note, again, that

S represents a certain event

0 an impossible event

A an event non - A

A  $\bigcap$  B = 0 = indicates that A and B are mutually exclusive (no elements in common)

AUB represents event A or B

ANB represents event A and B

A set function P(A) shall be called a probability measure on  $\beta$  if it satisfies the following axioms:

 $\text{(P$_1$)} \quad \text{For every A in $\beta$} \quad \text{, its value is a non-negative real}$  number  $\text{P(A)} \geq \text{O} \quad \text{for } \text{A} \in \beta$ 

$$(P_2) \quad P(S) = 1$$

(P<sub>3</sub>) If  $A_1$ ,  $A_2$ , ...  $A_j$ , ... is a finite or countable sequence of mutual events, then

$$P\left(\bigcup_{(j)} A_{j}\right) = \sum_{(j)} P(A_{j})$$

For  $A \in \mathcal{B}$  , the designation P(A) will be called the probability of the event A. Note, again, that

a. 
$$P(A) + P(A) = 1$$
 (to prove this use  $P_3$  and  $P_2$ )

b. 
$$0 \le P(A) \le 1$$
 for any event A (from a)

c. 
$$P(\theta) = 0$$
 [from S  $U \theta = S \rightarrow P(S) + P(\theta) = P(S)$ ]

#### 2. <u>Probability Distribution</u>

When a random experiment is studied, one would like to know the set of all its possible outcomes (outcome space), the subsets of this set to which probabilities are assigned (events) and, finally, the probabilities assigned to these subsets.

A probability distribution is a triple (S,  $\beta$ , P) where S is an outcome space,  $\beta$  a Borel field on S, and P a probability measure on  $\beta$ , hence, in order to know a probability distribution, each of its three components S,  $\beta$ , and P must be given.

#### 3. Continuous Probability Distributions and Probability Density

Let  $\mathbf{R}_n$  denote the n - dimensional Euclidean space.

A point in R  $_n$  is an n - tuple of coordinates (X  $_1$  , X  $_2$  ... X  $_n$  ) and can be represented in the abbreviated notation:

$$(X_1, X_2, ... X_n) = X$$

In  $R_1$  (Line), any set defined by a  $\leq X \leq b$ , where a and b are finite real numbers, is called a closed interval.

In  $R_2$  (plane), sets of the form,  $a \le X_1 \le b$ ,  $c \le X_2 \le d$  are called closed rectangles. In general, then, for any number of dimensions  $n \ge 1$ , sets defined by  $a_1 \le X_1 \le b_1$ ,  $a_2 \le X_2 \le b_2$ , ...  $a_n \le X_n \le b_n$  will be called closed n-dimensional intervals.

A probability distribution (S,  $\beta$ , P) is called <u>continuous</u> when S is a Euclidean space  $R_n$ ,  $\beta$  a Borel field containing all open and closed intervals in  $R_n$  and P is defined as follows:

If a function f:

$$f(X_1, X_2,...X_n) = f(X)$$

has the following properties:

(C<sub>1</sub>) 
$$f(X) \ge 0$$
 for all  $X \in R_n$   
(C<sub>2</sub>)  $\int_A f(X) dX$  exists for all  $A \in \beta$ 

$$(C_3) \quad \int_{R_n} f(X) \, dX = 1$$

Then, the probability measure P is defined as

$$P(A) = \int_A f(X) dX$$
 for every A

and the function f satisfying  $C_1$ ,  $C_2$ ,  $C_3$  is called a probability density.

The integrals in  $C_2$  and  $C_3$  are n-dimensional integrals, and dX denotes the n-dimensional volume element.

Remarks: The Borel field,  $\beta$  in  $R_n$ , contains all open and closed intervals, and all elementary geometric regions, such as interiors of closed polygons, curves in the plane, and solids in  $R_3$ , with their boundaries included or excluded. So that

$$P(A) = \int_{A} f(X) dX$$
 (29)

ascribes a probability to any such elementary geometric region. Furthermore, every bounded set  $A \in \mathcal{B}$  has a volume V(A) which, for all elementary geometric regions, is equal to the usual volume of those regions.

One can also show that (29) assigns probability 0 to any geometric region which has n- dimensional volume equal to zero. In particular, no matter what the probability density f(X), probability zero will be assigned to single points or finite sets of points in  $R_1$ , to arcs of curves or polygons in  $R_2$ , to two-dimensional surfaces or pieces of such surfaces in  $R_3$ , etc.

NOTE: Any function, g(X) on  $R_n$ , which has the properties  $C_1$  and  $C_2$  and such that

$$I = \int_{R_0} g(X) dX \neq 0$$
 (30)

may be used to obtain a probability density in the form:

$$f(X) = Kg(X)$$

for it follows from  $C_1$  and that I > 0, hence, writing K = 1/I, we have

$$f(X) = (\frac{1}{I}) g(X) = K g(X) \ge 0$$
 for all X; from  $C_2$ 

$$\int_{A} f(X) dX = K \int_{A} g(X) dX \text{ exists for all } A \in B$$

and

$$\int_{R_n} f(X) dX = \frac{1}{I} \int_{R_n} g(X) dX = 1$$
 (31)

so that f(X) satisfies  $C_1$ ,  $C_2$ ,  $C_3$ . The constant K = 1/I is called the normalizing constant.

#### 4. Uniform Distribution

Let  ${\tt I}{\tt I}$  be any domain in  ${\tt R}_{\tt n}$  with finite positive volume:

$$0 < V(D)$$
,  $V < + \infty$ 

The function

$$g(X) = \begin{cases} 1 & \text{for } X \in D \\ 0 & \text{for } X \in \overline{D} \end{cases}$$

clearly has the property  $C_1$ . It also satisfies (30) since

$$\int_{R_{D}} g(X) dX = \int_{D} 1 dX = V$$

and for any set  $\mathbf{A} \in \boldsymbol{\beta}$  , we have

$$\int_{A} g(X) dX = \int_{A \cap D} g(X) dX + \int_{A \cap \overline{D}} g(X) dX = \int_{A \cap D} 1 dX = V(A \cap D)$$

which is finite since it is  $\leq$  V(D). By using the normalizing constant

$$K = 1 / V$$

we obtain the probability density.

$$f(X) = \begin{cases} 1 / V(D) & \text{for } X \in D \\ 0 & \text{for } X \in \overline{D} \end{cases}$$
 (32)

The continuous probability distribution on  $R_n$  with the probability density (32) is known as the uniform distribution on D.

#### 5. Cumulative Distribution Functions

For a one-dimensional random variable X, the function

$$F(S) = P(\{X : X \le S\}) = P(X \le S)$$

is called the cumulative distribution function of X or the distribution function (d.f.) of X.

or

F(S) is the probability of the event (the random variable X) which assumes a value less than or equal to S.

For a one-dimensional continuous random variable X with the probability density f(X):

a.

$$F(S) = \int_{-\infty}^{S} f(X) dX$$
 (33)

b. F(S) is a non-decreasing and continuous function of S, its derivative exists at every point of continuity of f and, at every such point, we have

$$\frac{dF(S)}{dS} = f(S) \tag{34}$$

c. For every pair of real numbers,  $S_1 \le S_2$ , the probability of the event  $\left\{S_1 < X \le S_2\right\}$  is

$$P(S_1 < X \le S_2) = F(S_2) - F(S_1)$$
 (35)

d. Also

$$\lim_{S \to -\infty} F(S) = 0$$

$$\lim_{S \to \infty} F(S) = 1$$

$$S \to \infty$$

## 6. Continuous n- Dimensional Random Variables

If (X, Y) has the joint probability density f(x,y), then the marginal probability densities of x and y are:

$$g(X) = \int_{-\infty}^{+\infty} f(x, y) dy$$

$$h(Y) = \int_{-\infty}^{+\infty} f(x, y) dx$$
(36)

#### 7. Constructing the Model

Implementing a simulation model requires random numbers in order to obtain random observations from probability distributions.

 $\underline{\underline{\text{The first step}}}$  is to construct the cumulative distribution function

$$F(x) = P[X \le x]$$

where x is the random variable involved. This can be done by writing the equation for this function, or graphically plotting the function, or by developing a table giving the value of x for uniformly spaced values of F(x) from 0 to 1.

The second step is to generate a random decimal number between 0 and 1. This is done by obtaining a random integer number having the desired number of digits (including any leading zeros) and then placing a decimal point in front of it.

The final step is to set  $P(X \le x)$  equal to the random decimal number and solve for x. This value of X is the desired random observation from the probability function (Figure 3).

The following procedure is recommended for locating the major axis of the fibers in a cylindrical shaped model (Figure 4):

- 1. Determine  $r \rightarrow$  the distance from the center of the base of the cylinder (0  $\leq r \leq R$ ).
- 2. Determine  $\theta$  the angle or location of a point on the base  $(0 \le \theta \le 2\pi)$ .
  - 3. Determine  $z \rightarrow$  the height of the end point within the cylinder.
  - 4. Introduce length of fiber ₹.
- 5. Determine  $\alpha$  the angular direction of fiber in a plane parallel to the xy-plane (0  $\leq \alpha \leq 2\pi$ ).
- 6. Determine  $\phi$  the angular direction relative to the xy-plane  $(0 \le \phi \le 2\pi)$ .

NOTE: 5 Random numbers are needed.

a. To generate random observations on the base of the cylinder. From the uniform distribution, the joint probability density function

$$f(x,y) = \begin{cases} \frac{1}{\pi R^2} & \text{in } x^2 + y^2 = R^2 \\ 0 & (37) \end{cases}$$

elsewhere (e.w.)

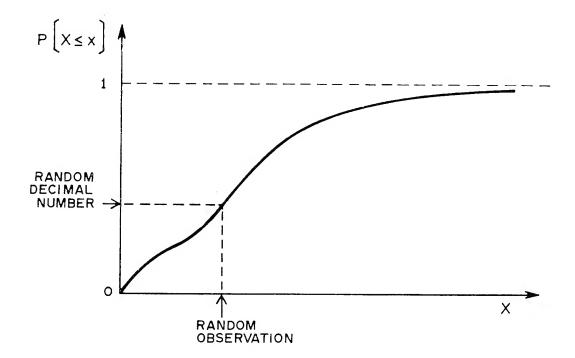


Figure 3. Illustration of Procedure for Obtaining a Random Observation From a Given Cumulative Distribution Function.

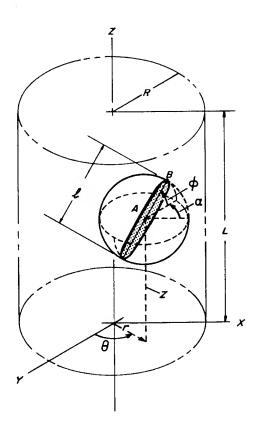


Figure 4. A Fiber in a Cylindrical Shaped Model.

transforms 
$$f(x, y)$$
 into  $g(r, \theta)$  let  $x = r \cos \theta$   
 $y = r \sin \theta$ 

$$g(r, \theta): f(x, y) \left| \frac{\partial(x, y)}{\partial(r, \theta)} \right|$$

$$\left| \frac{\partial(x, y)}{\partial(r, \theta)} \right| = \begin{vmatrix} \cos \theta & \sin \theta \\ -r \sin \theta & r \cos \theta \end{vmatrix} = r$$

or  $g(r, \theta) = \frac{r}{\pi R^2}$ 

The marginal probability densities of r and 0 are:

$$f(r) = \int_{0}^{2\pi} \frac{r}{\pi R^{2}} d\theta = \frac{2r}{R^{2}} \quad \text{where} \quad 0 \le r \le R$$

and

$$L(\theta) = \int_0^R \frac{r}{\pi R^2} dr = \frac{1}{2\pi}$$
 where  $0 \le \theta \le 2\pi$ 

The cumulative distribution functions of f(r) and  $L(\theta)$  are:

$$G(r) = \int_{0}^{r} \frac{2r}{R^{2}} dr = \frac{r^{2}}{R^{2}} \qquad \text{where} \qquad 0 \le r \le R$$

$$G(\theta) = \int_{0}^{\theta} \frac{1}{2\pi} d\theta = \frac{\theta}{2\pi} \qquad \text{where} \quad 0 \le \theta \le 2\pi$$

To generate random observations, see final step in paragraph 7, Constructing the Model.

Let 
$$x = \frac{r^2}{R^2}$$
, then

$$\mathbf{r} = R\sqrt{x} \qquad \text{or}$$

$$G^{-1}(\mathbf{x}) = R\sqrt{x} \qquad 0 \le \mathbf{x} \le 1$$
(38)

Similarly, let

$$y = \frac{\theta}{2\pi}$$
 then  $\theta = 2\pi y = F^{-1}(y)$ 

then

$$\theta = F^{-1}(u) = 2\pi u \qquad 0 \le u \le 1$$
 (39)

where x and u are different random numbers.

b. To generate random observations inside the cylinder. From the uniform distribution, the joint probability density function (for distributing points on a sphere)

$$f(x,y,z) = \begin{cases} \frac{1}{4/3 \pi R^3} & \text{in } x^2 + y^2 + z^2 \\ 0 & \text{e.w.} \end{cases}$$
 (40)

let

$$x = r \sin \phi \cos \theta$$
  
 $y = r \sin \phi \sin \theta$   
 $z = r \cos \phi$ 

then

$$g(r, \phi, \theta) : f(x,y,z) \left| \frac{\partial (x,y,z)}{\partial (r,\phi,\theta)} \right|$$

$$\begin{vmatrix} \frac{\partial(x, y, z)}{\partial(r, \phi, \theta)} \end{vmatrix} = \begin{vmatrix} \sin\phi & \cos\theta & \sin\phi & \sin\theta & \cos\phi \\ r\cos\phi & \cos\theta & r\cos\phi & \sin\theta & -r\sin\phi \\ -r\sin\phi & \sin\theta & r\sin\phi & \cos\theta & 0 \end{vmatrix}$$
$$= r^2 \sin\phi$$

or

$$g(r, \phi, \theta) = \frac{3}{4\pi R^3}$$
  $r^2 \sin \phi$ 

then

$$g(\mathbf{r}, \boldsymbol{\phi}, \boldsymbol{\theta}) = \begin{cases} \frac{3 \mathbf{r}^2 \sin \boldsymbol{\phi}}{4 \pi \mathbf{R}^3} & \inf \begin{cases} 0 \le \theta \le 2\pi \\ 0 \le \boldsymbol{\phi} \le \pi \\ 0 \le \mathbf{r} \le \mathbf{R} \end{cases} \end{cases}$$

The marginal probability densities of r, $\phi$  and  $\theta$  are:

$$h(r) = \int_0^{\pi} \int_0^{2\pi} \frac{3 r^2 \sin \phi}{4 \pi R^3} d\theta d\phi = \frac{3 r^2}{R}$$

or

$$h(r) = \begin{cases} \frac{3 r^2}{R^3} & \text{in } 0 \le r \le R \\ 0 & \text{e.w.} \end{cases}$$

$$g(\phi) = \int_0^{2\pi} \int_0^R \frac{3 r^2 \sin \phi}{4 \pi R^3} dr d\theta = \frac{\theta \sin \phi}{4 \pi}$$

or

$$g(\phi) = \begin{cases} \frac{\sin \phi}{2} & \text{in } 0 \le \phi \le \pi \\ 0 & \text{e.w.} \end{cases}$$

$$f(\theta) = \int_0^{\pi} \int_0^R \frac{3 r^2 \sin \phi}{4 \pi R^3} dr d\phi = \frac{1}{2 \pi}$$

$$f(\theta) = \begin{cases} \frac{1}{2\pi} & \text{in } 0 \le \phi \le 2\pi \\ 0 & \text{e.w.} \end{cases}$$

The cumulative distribution functions of h(r),  $g(\phi)$ , and  $f(\theta)$  are:

$$H(r) = \int_{0}^{r} \frac{3 r^{2}}{R 3} dr = \frac{r^{3}}{R^{3}}$$

$$G(\phi) = \frac{1}{2} \int_0^{\phi} \sin \phi \, d\phi = \frac{1 - \cos \phi}{2}$$

$$f(\theta) = \int_0^{\theta} \frac{1}{2\pi} d\theta = \frac{\theta}{2\pi}$$

To generate random observations:

for 
$$H(r) \rightarrow 1$$
et  $C = \frac{r^3}{R^5} \rightarrow r = R \sqrt[3]{C}$ 

$$r = C^{-1}(u) = R \sqrt[3]{u} \qquad 0 \le u \le 1 \qquad (41)$$

for 
$$G(\phi)$$
 — let  $b = \frac{1 - \cos \phi}{2} - \cos \phi = 1 - 2b$ 

$$\alpha = \phi = \cos^{-1} (1 - 2b) = B^{-1} (u)$$

$$\alpha = B^{-1} (u) = \cos^{-1} (1 - 2u) \quad 0 \le u \le 1$$
 (42)

for 
$$F(\theta)$$
 - let  $a = \frac{\theta}{2\pi}$  then  $\theta = 2\pi\sigma = A^{-1}(\theta)$ 

$$\phi = A^{-1}(u) = 2\pi u \qquad 0 \le u \le 1 \qquad (43)$$

where u is a random number.

Equations (38), (39), (41), (42), and (43) are used with the length of the fiber to locate the axis of the fiber in space.

Once this axis has been located, its position in space has to be established by introducing a new coordinate system.

c. Rotation of axis in three dimensions. From reference 23,

$$x' = \ell_1 x + m_1 y + n_1 z$$

$$y' = \ell_2 x + m_2 y + n_2 z$$

$$z' = \ell_3 x + m_3 y + n_3 z$$
(44)

where ( $\{1, m_1, m_1, \ldots, m_3\}$ ) are the cosines of the angles between 0x' and 0x, 0x' and 0y, ..., 0z' and 0z. The angles are in the direction of increasing  $x_i$  to increasing  $x'_j$ , where  $(x_1, x_2, x_3)$  corresponds to (x, y, z), and  $(x'_i, x'_z, x_3')$  corresponds to (x', y', z'). It is now necessary to obtain  $\{1, m_i, m_i, m_i\}$  for i = 1, 2, 3 in order to locate the new coordinate axes X', Y', and Z'.

Let

$$l_{i} = \cos \alpha_{i}$$

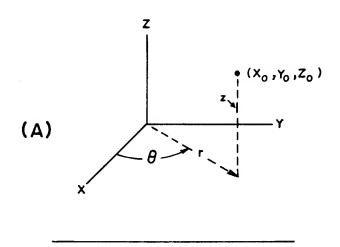
$$m_{i} = \cos \beta_{i}$$

$$n_{i} = \cos \gamma_{i}$$
for  $i = 1, 2, 3$ 

$$(45)$$

Recall that in paragraph 8, Constructing the Model,  $(\theta, r, z)$  were generated to obtain  $(x_0, y_0, z_0)$  which is the center of the new coordinate system X', Y', and Z'(Figure 5a).

(1) To find X': Recall also that the angles  $\alpha$  and  $\phi$  and the length of the fiber L were generated; these three parameters  $(\alpha, \phi, L)$  are used to locate  $(x_1, y_1, z_1)$ , and to obtain  $\cos \alpha_1$ ,  $\cos \beta_1$ , and  $\cos \gamma_1$ , (Figure 5b)



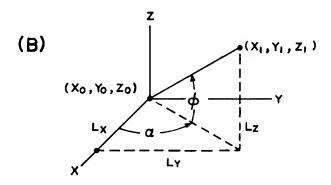


Figure 5. Location of the Center and End Point of a Fiber.

Then

$$L_{x} = L \cos \phi \cos \alpha = x_{1} - x_{0}$$

$$L_{y} = L \cos \phi \sin \alpha = y_{1} - y_{0}$$

$$L_{z} = L \sin \phi = z_{1} - z_{0}$$
(46)

or

$$\cos \alpha_1 = \frac{L_x}{L}, \cos \beta_1 = \frac{L_y}{L}; \cos \gamma_1 = \frac{L_z}{L}$$
 (47)

(2) To find Y' (Figure 6a): The equation of the plane perpendicular to a vector through  $(\mathbf{x_0},\,\mathbf{y_0},\,\mathbf{z_0})$  and  $(\mathbf{x_1},\,\mathbf{y_1},\,\mathbf{z_1})$  is

$$(x_1 - x_0) (x_2 - x_0) + (y_1 - y_0) (y_2 - y_0) + (z_1 - z_0) (z_2 - z_0) = 0$$
 (48)

Y' is chosen so that it is always parallel to the x-y plane; hence  $z_2 = 0$  and (48) becomes

$$(x_1 - x_0) (x_2 - x_0) + (y_1 - y_0) (y_2 - y_0) = 0$$
 (49)

Let the distance between  $(x_0, y_0, z_0)$  and  $(x_2, y_0, z_2)$  be  $\ell = b$ ; then

$$(x_2 - x_0)^2 + (y_2 - y_0)^2 = \ell^2$$
 (50)

From (2) and (3):

$$x_2 - x_0 = \frac{-(y_1 - y_0) - (y_2 - y_0)}{(x_1 - x_0)}$$
 (51)

for  $x_1 \neq x_0$ 

Substituting now (51) into (50):

$$\frac{(y_1 - y_0)^2 (y_2 - y_0)^2}{(x_1 - x_0)^2} + (y_2 - y_0)^2 = \begin{cases} 2 \\ \end{cases}$$

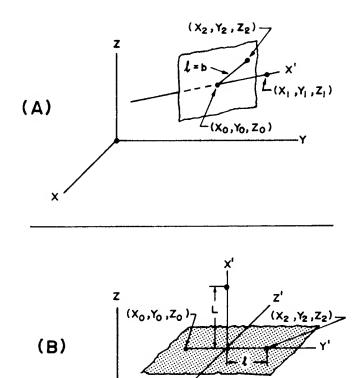


Figure 6. Rotation of Axes in Three Dimensions.

or

$$(y_2 - y_0) = \frac{(x_1 - x_0)!}{\sqrt{(x_1 - x_0)^2 + (y_1 - y_0)^2}} = \frac{L_x!}{\sqrt{L_x^2 + L_y^2}}$$
(52)

If  $x_1 = x_0$  and  $y_1 = y_0$ , then  $\phi = 90^\circ$ ; hence  $x_2 = x_0$ , and  $y_2 = y_0 + \ell$ (i.e., the Y and Y' are the same) (Figure 6b).

If  $x_1 = x_0$  and  $y_1 = y_0$ , then  $\theta = 90^\circ$ ; hence  $y_2 = y_0$  and  $x_2 = x_0 - 1$ . Thus,

$$\cos \alpha_2 = \frac{x_2 - x_0}{l}; \cos \beta_2 = \frac{y_2 - y_0}{l}; \cos \gamma_2 = \frac{z_2 - z_0}{l}$$
 (53)

where

$$\ell = \sqrt{(x_2 - x_0)^2 + (y_2 - y_0)^2 + (x_2 - z_0)^2}$$

(3) To find Z': Knowing X' and Y', Z' can be found by using the cross products:

$$Z' = X' \times Y'$$

$$= [(x_1 - x_0) \overline{i} + (y_1 - y_0) \overline{j} + (z_1 - z_0) \overline{k}] \times$$

$$[(x_2 - x_0) \overline{i} + (y_2 - y_0) \overline{j} + (z_2 - z_0) \overline{k}]$$
(54)

$$= [(y_1 - y_0) (z_2 - z_0) - (z_1 - z_0) (y_2 - y_0)] \overline{i} + [(z_1 - z_0) (x_2 - x_0) - (x_1 - x_0) (z_2 - z_0)] \overline{j} + [(x_2 - x_0) (y_2 - y_0) + (y_1 - y_0) (x_2 - x_0)] \overline{k}$$

From preceding paragraph (2),  $z_2 = z_0$ , hence

$$z' = -(z_1 - z_0) (y_2 - y_0) i + (z_1 - z_0) (x_2 - x_0) j + (x_1 - x_0) (y_2 - y_0) - (y_1 - y_0) (x_2 - x_0) \overline{k}$$

$$Z' = c_1 \overline{i} + c_2 \overline{j} + c_3 \overline{k}$$
 (55)

where

$$c_1 = x_3 - x_0$$
,  $c_2 = y_3 - y_0$ ;  $c_3 = z_3 - z_0$ 

Then

$$\cos \alpha_3 = \frac{c_1}{d}; \cos \beta_3 = \frac{c_2}{d}, \cos \gamma_3 = \frac{c_3}{d} \tag{56}$$

where

$$d = \sqrt{c_1^2 + c_2^2 + c_3^2}$$

Paragraphs 8c (1), (2), and (3) give the new coordinate system (X', Y', Z'), with the center at  $(x_0, y_0, z_0)$ , and the following nine angles between (X, Y, Z) and (X', Y', Z'):

$$\cos \alpha_{1} = \frac{x_{1} - x_{0}}{L}; \cos \beta_{1} = \frac{y_{2} - y_{0}}{L}; \cos \gamma_{1} = \frac{z_{1} - z_{0}}{L}$$

$$\cos \alpha_{2} = \frac{x_{2} - x_{0}}{L}; \cos \beta_{2} = \frac{y_{2} - y_{0}}{L}; \cos \gamma_{2} = 0$$

$$\cos \alpha_{3} = \frac{x_{3} - x_{0}}{d}; \cos \beta_{3} = \frac{y_{3} - y_{0}}{d}; \cos \gamma_{3} = \frac{z_{3} - z_{0}}{d}$$
(57)

where

$$L = [(x_1 - x_0)^2 + (y_1 - y_0)^2 + (z_1 - z_0)^2]^{1/2}$$

$$\begin{cases} l = [(x_2 - x_0)^2 + (y_2 - y_0)^2 + (z_2 - z_0)^2]^{1/2} \\ l = [(x_3 - x_0)^2 + (y_3 - y_0)^2 + (z_3 - z_0)^2]^{1/2} \end{cases}$$

d. Locating fibers uniformly in a cylindrical volume. To generalize the fiber's geometry, it is assumed that the fiber has an elliptical cross section. Then, from (57), L = a,  $\frac{1}{4} = b$ , and d = c are

the distances from the center of the ellipsoid  $(x_0, y_0, z_0)$  to the major vertex and minor vertexes respectively.

To place an ellipsoid or fiber in space, it is necessary to generate  $\theta$ , r, z, x, and  $\phi$  according to a predetermined distribution function, and then calculate the nine angles describing the relationship of the fiber or ellipsoid to the X, Y, Z axis.

The general equation of an ellipsoid is developed next.

The equation of an ellipsoid centered at the origin, assuming no translations or rotations, is

$$\frac{(x'')^2}{a^2} + \frac{(y'')^2}{b^2} + \frac{(z'')^2}{c^2} = 1$$
 (58)

where 2a is the length of the major axis, and 2b = 2c the length of the minor axis.

Including rotations from equation (44), and the translations:

$$x' = x - h$$

$$y' = y - j$$

$$z' = z - k$$
(59)

The general equation of an ellipsoid, with respect to the X, Y, Z axis, becomes:

$$\frac{(l_1 x' + m_1 y' + n_1 z')^2}{a^2} + \frac{(l_2 x' + m_2 y' + n_2 z')^2}{b^2} + \frac{(l_3 x' + m_3 y' + n_3 z')^2}{c^2} = 1$$
(60)

where x', y', z' are given in (59), and (h, j, k) is equivalent to  $(x_0, y_0, z_0)$ .

Once the fibers are generated, they have to be placed in a predetermined volume (in this study, a cylindrical volume) to simulate, for instance, an experimental specimen. To obtain a realistic specimen, with uniformly distributed fibers, it is necessary to reject any fiber which intersects another fiber (i.e., a fiber cannot occupy the volume of another one) or intersects the specimen's boundary.

e. Method of finding the intersection of two fibers. Assuming two ellipsoidal fibers are given with centers at  $(h_1, j_1, k_1)$  and  $(h_2, j_2, k_2)$ , let a > b, and a > c, then the following two conditions must be satisfied before the two fibers are checked for an intersection:

$$[(h_2 - h_1)^2 + (j_2 - j_1)^2 + (k_2 - k_1)^2]^{1/2} < 2a$$
 (61)

and

$$[(h_2 - h_1)^2 + (j_2 - j_1)^2 + (k_2 - k_1)^2]^{1/2} \ge 2c$$
 (62)

NOTE: If  $[(h_2 - h_1)^2 + (j_2 - j_1)^2 + (k_2 - k_1)^2]^{1/2} \ge 2a$ , there is no possible way for the fibers to intersect, and the second fiber is kept. If  $[(h_2 - h_1)^2 + (j_2 - j_1)^2 + (k_2 - k_1)^2] < 2c$ , then the two fibers will intersect. The second fiber is discarded, and another one is generated.

When another fiber is generated, assuming that it satisfies conditions (61) and (62), the following procedure is used to determine if the fibers intersect:

Represent the fibers as vectors  $\overline{V}_1$  and  $\overline{V}_2$  (Figure 7), where  $\overline{V}_1 = a_1 \overline{i} + b_1 \overline{j} + c_1 \overline{k}$  (63)

and

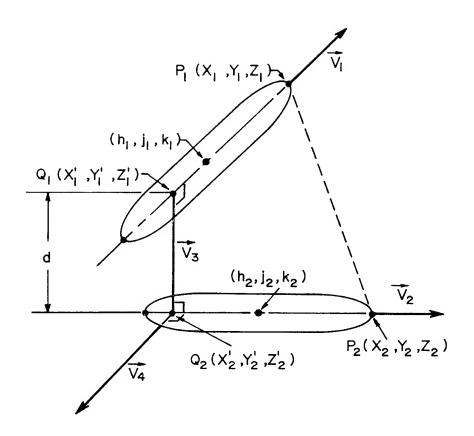


Figure 7. Representation of Two Random Fibers.

$$\overline{V}_2 = a_2\overline{i} + b_2\overline{j} + c_2\overline{k} \tag{64}$$

Let  $P_1$  (x<sub>1</sub>, y<sub>1</sub>, z<sub>1</sub>) and  $P_2$  (x<sub>2</sub>, y<sub>2</sub>, z<sub>2</sub>) be the end points of fibers  $\overline{V}_1$  and  $\overline{V}_2$  respectively;  $P_1$  and  $P_2$ , then, determine the vector  $\overline{P_1P_2}$ .

In plane geometry, nonparallel lines intersect. In solid geometry, however, it is possible to have nonparallel lines which do not intersect. In the following analysis, the perpendicular distance, d, between nonparallel lines is investigated. If the perpendicular distance, d, between the two vectors containing the fibers is zero, then  $\overline{V}_1$  and  $\overline{V}_2$  intersect; if  $d\neq 0$ , but is less than the minor axis of the fibers (i.e., b anc c), then  $\overline{V}_1$  and  $\overline{V}_2$ , or the fibers, are "skew".

In the latter case, the coordinates of the end points of the perpendicular distance must be found to actually determine if the fibers do intersect. That is, if the coordinates of the intersections lie within the end points of both fibers, the fibers intersect; otherwise, there is no intersection.

The perpendicular distance between  $\overline{\mathrm{V}}_1$  and  $\overline{\mathrm{V}}_2$  is

$$d = \left| \overline{P_1} \, \overline{P_2} \, \cdot \, \frac{\overline{V_1} \, x \, \overline{V_2}}{\left| \overline{V_1} \, x \, \overline{V_2} \right|} \right| \tag{65}$$

The coordinates of d are found next.

Let a plane, p, pass through the point  $\mathbb{Q}_2$   $(\mathbf{x}_2,\,\mathbf{y}_2,\,\mathbf{z}_2)$ , and let the vector  $\overline{\mathbb{V}}_4$  be normal to p:

$$\vec{V}_4 = \vec{V}_2 \times \vec{V}_3 = a_4 \vec{i} + b_4 \vec{j} + c_4 \vec{k}$$
 (66)

where

$$\overline{V}_3 = \overline{V}_1 \times \overline{V}_2 \tag{67}$$

Since the vector  $\overline{\mathbf{P}_2}$   $\mathbf{Q}_2$  is perpendicular to  $\overline{\mathbf{V}}_4$ , then

$$\overline{V}_4$$
 .  $\overline{P}_2 \overline{Q}_2 = 0$ 

hence

$$(a_4\bar{i} + b_4\bar{j} + c_4\bar{k}) \cdot [(x_2 - x_2')\bar{i} + (y_2 - y_2')\bar{j} + (z_2 - z_2')\bar{k}] = 0$$

or

$$a_4 (x_2 - x_2') + b_4 (y_2 - y_2') + c_4 (z_2 - z_2') = 0$$

The equation of the plane, p, can be rewritten by multiplying and transposing terms not containing the variables:

$$a_4 x_2 + b_4 y_2 + c_4 z_2 = a_4 x_2' + b_4 y_2' + c_4 z_2'$$

or

$$a_4 x_2 + b_4 y_2 + c_4 z_2 = D$$
 (68)

The symmetric form equations for a straight line which passes through  $P_1$  ( $x_1$ ,  $y_1$ ,  $z_1$ ), having the direction  $\overline{V}_1$ , can be written in the form:

$$\frac{x_1 - x_1'}{a_1} = \frac{y_1 - y_1'}{b_1} = \frac{z_1 - z_1'}{c_1} \tag{69}$$

The simultaneous solution of equations (68) and (69) yield the coordinates of  $\mathbb{Q}_1$ .

The coordinates of  $Q_2$  are found as follows:

A unit vector in the direction of  $\overline{\mathbb{Q}_1}$   $\mathbb{Q}_2$  is  $\overline{\mathbb{Q}_3}$  since  $\overline{\mathbb{Q}_1}$   $\mathbb{Q}_2$  has a length, d, then

$$\overline{V}_5 = \overline{Q}_1 \overline{Q}_2 = d \frac{\overline{V}_3}{|V_3|} = a_5\overline{i} + b_5\overline{j} + c_5\overline{k}$$
 (70)

or

$$\overline{Q_1} \overline{Q_2} = (x_1' - x_2')\overline{i} + (y_1' - y_2')\overline{j} + (z_1' - z_2')\overline{k}$$

Then, the solution set  $(x_2', y_2', z_2')$  is obtained by

$$x_{2}' = x_{1}' - a_{5}$$

$$y_{2}' = y_{1}' - b_{5}$$

$$z_{2}' = z_{1}' - c_{5}$$
(71)

Equations (64), (68), (69) and (71) are then used to determine if two fibers intersect.

- f. Method for determining whether or not a fiber is entirely contained within the prescribed cylindrical model. The parts to be considered are the top and bottom of the cylinder and the cylindrical envelope itself.
- (1) If the z distance of the end points of the fiber generated is less than the length (L) of the model and greater than zero, the fiber is accepted; otherwise, it is rejected.
- (2) If the x or y distance of the end points of the fiber generated is less than the radius (R) of the model, the fiber is accepted; otherwise, it is rejected.

#### 8. Thermal Model

Once the fibers have been placed in the cylindrical volume, a method has to be devised to obtain the thermal conductivity of the composite model. The first step is to subdivide the system into a number

of small but finite subvolumes, and assign a reference number to each.

The second step consists of writing heat-balance equations for each subvolume and then generalize the results.

The simplest case of a one-dimensional heat flow, namely heat conduction through a subvolume, will be treated. It is assumed that the system is exposed to a high temperature medium, i.e., a heat source of known and constant temperature on one side and a low-temperature medium, i.e., a heat sink of known and constant temperature on the other side. Each subvolume is assumed to have an isothermal boundary at face A (Figure 8), (i.e., since  $T_2 = T_3 = T_a$ , heat can only flow along the fictitious channel) and on insulated surface  $S_1$ , (i.e., no heat is transferred from  $S_1$  to  $S_2$  or  $S_3$ ).

If the line,  $z_1$ , is dropped through the center of the subvolume, it will intersect the fibers at points 1, 2, 3, etc.. Once these points are found, the channel is further simplified as shown in Figure 8, and the steady-state heat conduction equation can now be applied.

$$Q_1 = \frac{K_{B_1} A_1 \Delta T}{I}$$
 (72)

where

A = channel cross-sectional area

$$\Delta T = T_A - T_B$$

 $K_{\mathbf{B}_1}$  = Thermal conductivity of channel 1

L = length of channel and/or model

 $L_{f}$  = length of fiber material

 $L_{m}$  = length of matrix material

Let

$$Q'' = \frac{Q'}{\Delta} = \frac{K_{B_1} \Delta T}{I} = constant$$

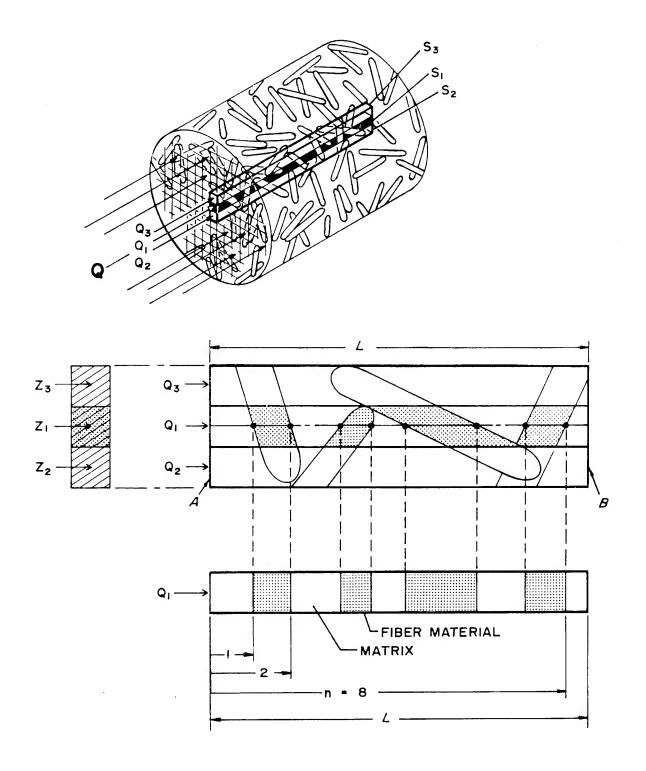


Figure 8. Thermal Model Used in This Study.

then

$$(\Delta T) \text{ matrix} = \frac{Q''}{K_{m}} [(L_{m})_{1} + (L_{m})_{2} + \cdots + (L_{m})_{n}]$$

$$= \frac{Q''}{K_{m}} \sum_{n=1}^{P} (L_{m})_{n}$$
(73)

$$(\Delta T) \text{ fiber} = \frac{Q''}{K_f} \qquad \sum_{n=1}^{P} (L_f)_r$$
 (74)

or

$$\Delta T = \frac{Q''}{K_{m}} \qquad \sum_{n=1}^{P} (L_{m})_{n} + \frac{Q''}{K_{f}} \qquad \sum_{m=1}^{r} (L_{f})_{m}$$

then

$$\frac{\Delta T}{Q''} = \frac{1}{K_{m}} \sum_{n=1}^{P} (L_{m})_{n} + \frac{1}{K_{f}} \sum_{m=1}^{r} (L_{f})_{m} = \frac{L}{K_{B_{1}}}$$

which can be rewritten as:

$$\frac{1}{K_{B}} = \frac{1}{L} \left[ \frac{1}{K_{m}} \sum_{n=1}^{P} (L_{m}) n + \frac{1}{K_{f}} \sum_{m=1}^{r} (L_{f})_{m} \right]$$
 (75)

Let  $V_{\mbox{\scriptsize R}_{1}}$  be the volume fraction of the fiber material, then

$$V_{R_1} = \frac{1}{L} \sum_{m=1}^{r} (L_f)_m$$
 (76)

note that  $V_{R_1} = 1$  when  $\sum_{m} (L_f)_m = L$ 

$$\frac{1}{K_{B1}} = \frac{V_{R_1}}{K_f} + \frac{1 - V_{R_1}}{K_m}$$
 (77)

Knowing then  $V_{R_1}$ ,  $K_f$ , and  $K_m$ ,  $K_{B_1}$  can be found.

The same procedure applies to the other channels. When  $K_{B_1}$ ,  $K_{B_2}$ ,--- $K_{B_n}$  are found, the overall average,  $K_B$ , of the model can be obtained.

- a. To find fiber material in each flow channel. For various lines parallel to the z axis from z = 0 to z = L, it is desired to determine the points of intersection between these lines and the ellipsoids. The base circle of the model,  $x^2 + y^2 = R^2$ , is divided into N parts (each part should be  $\leq$  the cross sectional area of the given fiber), with lines going through the center of each part (Figure 8). The z's denote the lines. To determine the intersection of a line with an ellipsoid, we proceed as follows: assume a particular line has x and y coordinates of x and y. This line has to be solved with every ellipsoid in the direction of the heat flow (z direction). If there is no solution, there is no intersection. If there is a solution  $\overline{z}_1$ ,  $\overline{z}_2$ ,  $|\overline{z}_2 \overline{z}_1|$  is saved. This is done for all lines within the circle.
- b. <u>Intersection of flow lines with fibers</u>. Equation (63) is solved for z:

$$z = \frac{-\bar{b} \pm \sqrt{\bar{b}^2 - 4\bar{a}\bar{c}}}{2\bar{a}}$$
 (78)

where

$$\bar{a} = C$$

$$\bar{b} = Ex + Fy$$

$$\bar{c} = A x^2 + By^2 + Dxy + Gx + Hy + I$$

In this study, H = G = I = 0

Solving now (60) for z' in terms of x' and y', the following values for a, b, and c are obtained:

$$a = b^{2} c^{2} n_{1}^{2} + a^{2} c^{2} n_{2}^{2} + a^{2} b^{2} n_{3}^{2}$$

$$b = 2 [n_{1} b^{2} c^{2} (l_{1}x' + m_{1}y') + n_{2} a^{2} c^{2} (l_{2}x' + m_{2}y') + n_{3} a^{2} b^{2} (l_{3}x' + m_{3}y')]$$

$$c = b^{2} c^{2} (l_{1}x' + m_{1}y')^{2} + a^{2} c^{2} (l_{2}x' + m_{2}y') + a^{2} c^{2} (l_{3}x' + m_{3}y') - a^{2} b^{2} c^{2}$$

$$(79)$$

A computer program is provided in Appendix A to find the overall average thermal conductivity,  $K_{\rm B}$ , when the following is given:

- a. Fiber geometry.
- b. Model geometry.
- c. Overall fiber volume ratio.
- d. Thermal conductivity of the fiber.
- e. Thermal conductivity of the matrix.

# B. <u>Discussion of the Planning and Analysis</u> of Comparative Experiment

- 1. General Consideration in Planning the Thermal Conductivity Experiment of Composite Materials
- The nature of experimentation. An experiment has been defined, in the most general sense, as "a considered course of action aimed at answering one or more carefully framed questions." In the study of Thermal Conductivity of Composite media, however, we are concerned with a more restricted kind of experiment in which the experimenter does something to at least some of the things under study and then observes the effect of his action. The things under study which are being deliberately varied in a controlled fashion may be called factors. These factors may be quantitative factors, such as temperature which can be varied along a continuous scale, or they may be qualitative factors, such as different compositions of matrix and fibers. The use of the proper experimental pattern aids in the evaluation of the factors. Many books have been written on the general principles of experimentation; the book by Wilson<sup>24</sup> is used in this analysis. There are certain characteristics an experiment obviously must have in order to accomplish anything at all:
  - (1) There must be a clearly defined objective:

and equipment.

- (a) Choice of factors, including their range.
- (b) Choice of experimental materials, procedure,
- (c) Knowledge of what the results are applicable to.

- (2) As far as possible, effects of factors should not be obscured by other variables. The use of an appropriate experimental pattern helps to free the comparison of interest from the effects of uncontrolled variables and simplifies the analysis of the results.
- (3) As far as possible, the experiment should be free from bias. Some variables may be taken into account by planned grouping; for other variables, the use of randomization is desirable.
- (4) An experiment should provide a measure of precision (experimental error). Replication provides the measure of precision; note that randomization assures validity of the measure of precision.
- (5) The experiment must have sufficient precision to accomplish its purpose set forth in requisite (4). Greater precision may be achieved by refinements of the experimental pattern technique.
- b. Experimental pattern. A common experimental pattern is the so-called factorial design equipment, wherein we control several factors and investigate their effects at each of two or more levels. If two levels of each factor are involved, the experimental plan consists of taking an observation at each of the 2<sup>n</sup> possible combinations.
- c. <u>Planned grouping</u>. An important class of experimental patterns is characterized by planned grouping. This class is often called block designs. In engineering research, the tool of planned grouping can be used to take advantage of naturally homogeneous groupings in materials, machines, etc., or "background variables" which are not directly "factors" in the experiment. Statisticians have developed a variety of especially advantageous configurations of block designs, named and classified by their structure into randomized blocks, Latin

squares, incomplete blocks, lattices, etc., with a number of subcategories of each.

d. Randomization. Randomization is necessary to accomplish requisites (3) and (4) in preceding paragraph a. In order to eliminate bias from the experiment, experimental variables which are not specifically controlled as factors, or "blocked out" by planned grouping, should be randomized, e.g., the allocation of specimens to treatments or methods should be made by some mechanical method of randomization.

Randomization also assures valid estimates of experimental error and makes possible the application of statistical test of significance and the construction of confidence intervals.

"Randomization may be thought of as insurance and, like insurance, may sometimes be too expensive."

In general, we should try to think of all variables that could possibly affect the results, select as factors as many variables as can reasonably be studied, and use planned grouping where possible.

e. Replication. Replication (repetition) provides the measure of precision, an opportunity for the effects of uncontrolled factors to balance out, to aid randomization as a bias-decreasing tool, and to spot gross errors in the measurements.

### 2. Factorial and Fractional Factorial Experiments

a. Some general remarks. In the experimental study of the average conductivity  $(K_B)$  of composite materials, we are interested in investigating the effect of length  $(\cline{L})$ , cross section  $(a \times b)$  of the fiber, thermal conductivity of the fiber  $(K_f)$ , thermal conductivity of the matrix  $(K_m)$ , volume ratio of the fiber in the matrix  $(V_R)$ , on  $(K_B)$ ,

assuming a random distribution of fibers in the matrix.

The factors involved are then  $\ell$  , axb,  $K_f$ ,  $K_m$ , and  $V_R$ ; each specific value of the factors will be called a level.

In the past, one common experimental approach has been the so called "one at a time" approach. This kind of experiment would study the effect of varying "factors" one at a time, keeping the others constant. The results of such an experiment are fragmentary in the sense that we have learned about the effect of different levels of factors at one factor only and vice versa. In statistical language, there may be an "interaction" effect between the two factors within the range of interest, and the "one at a time" procedure does not enable us to detect it.

Experiment wherein we control several factors and investigate their effects at each of two or more levels. In the analysis of factorial experiments, we speak of main effects and interaction effects. Main effects of a given factor are always function of the average response at the various levels of the factor. In the case where a factor has two levels, the main effect is the difference between the responses at the two levels averaged over all levels of the other factors. If the difference in the response between two levels of factor A is the same regardless of the level of factor B (except for experimental error), we say that there is no interaction between A and B, or that AB interaction is zero.

If we have two levels of each of the factors A and B, then the AB interaction (omitting experimental error) is the difference in the responses of A at the second level of B minus the difference in the response of A at the first level of B. In general, if we have  $\underline{a}$  levels of

the factor A and  $\underline{b}$  levels of the factor B, then the AB interaction has (a - 1) (b - 1)

independent components.

For factorial experiments with three or more factors, interactions can be defined similarly. For instance, the ABC interaction is the interaction between the factor C and the AB interaction (or equally between the factor B and the AC interaction, or A and the BC interaction).

b. Internal estimates of errors. As in any experiment, we must have a measure of experimental error to use in judging the significance of the observed differences in treatments. In the larger factorial designs, estimates of higher-order interactions will be available. The usual assumption is that high-order interactions are physically impossible and that the estimates so labelled are actually estimates of experimental error. As a working rule, we often use third and higher-order interactions for error. The judgement of the experimenter will determine which interactions may reasonably be assumed to be meaningful and which may be assumed to be nothing more than error. These latter interactions may be combined to provide an internal estimate of error or a factorial experiment of reasonable size. For very small factorials, e.g., 2<sup>3</sup> or smaller, there are no estimates of high order interactions, and the experiment must be replicated in order to obtain an estimate of error from the experiment itself.

In the case of fractional factorials, there is obviously no point in replication of the experiment; further experimentation would probably be aimed at completing the full factorial or a larger fraction

of the full factorial.

c. Symbols. To identify each of the trials, in the factorial experiment of  $K_{\rm B}$ , the following notation is adapted:

A factor is identified by a capital letter, hence:

axb = B

 $K_f = C$ 

 $K_m = D$ 

 $V_{R} = E$ 

and the two levels of a factor by the subscripts: "zero" and "one". If we have five factors, A, B, C, D and E, then the corresponding levels of the factors ar  $A_0$ ,  $A_1$ ;  $B_0$ ,  $B_1$ ;  $C_0$ ,  $C_1$ ;  $D_0$ ,  $D_1$ ;  $E_0$ ,  $E_1$ ; respectively. By convention, the zero subscript refers to the lower level, to the normal condition, or to the absence of a condition, as appropriate. A trial is respresented by a combination of small letters denoting the levels of the factors in the trial. The presence of a small letter means that the factor is at the level denoted by the subscript 1. The absence of a letter means that the factor is at the level denoted by the subscript zero. Thus the symbol "a" represents the treatment combination where A is at level  $A_1$ , B is at level  $B_0$ , C is at level  $C_0$ , etc. The symbol "bc" represents the treatment combination where A is at  $C_1$ , etc.

Conventionally, the symbol (1) represents the treatment combination with each factor at its zero level. In our case, having five factors, each at two levels, then

$$2^n = 5 = 32$$

combinations, and the 32 trials, are represented by

(1)

a, b, ab,

c, ac, bc, abc,

d, ad, bd, abd, cd, acd, bcd, abcd,

e, ae, be, abe, ce, ace, bce, abce, de, ade, bde, abde, cde, acde, bcde, abcde.

- NOTE: 1. The treatment combinations are obtained by multiplying the new element by all previous treatment combinations.
- 2. The number of observations represents the average conductivity  $(K_{\mathrm{B}})$  obtained from a predetermined combination of factors.

Figure 9 summarizes the complete factorial experiment.

Explanation of Figure 9:

Sample 1 is equal to  $A_0$ ,  $B_0$ ,  $C_0$ ,  $D_0$ ,  $E_0$ 

Sample c is equal to  $A_0$ ,  $B_0$ ,  $C_1$ ,  $D_0$ ,  $E_0$ 

Sample abcde is equal to  $A_1$ ,  $B_1$ ,  $C_1$ ,  $D_1$ ,  $E_1$ 

The specimens used in this study are shown in Figures 10 and 11 where

			Δ	0		A <sub>I</sub>			
		B <sub>o</sub>		B <sub>I</sub>		Bo		B <sub>I</sub>	
		Co	Cı	Co	Ci	Co	CI	Co	C,
Do	Εo	1	С	b	bc	a	ac	ab	abc
	ΕĮ	e	ce	be	bce	ae	ace	abe	a p e
D,	Eo	đ	cd	bd	bcd	ad	acd		a p c q
	Ε <sub>I</sub>	de	cde	bde	<sup>b</sup> cd <sub>e</sub>	ade	a <sub>c</sub> de	abde	a <sub>bcde</sub>

Figure 9. Factorial Design With Five Factors at Two Levels.

		J				
		Ço				
	Во	5	0			
		Co	0			
		5				9
0	<b>~</b>	Co				
A	Bo	5			0	
		ပိ				
			E <sub>0</sub>	5 <u>n</u>	E	E E

Figure 10. Factorial Design With Five Factors at Two Levels (1/4 Inch Sample).

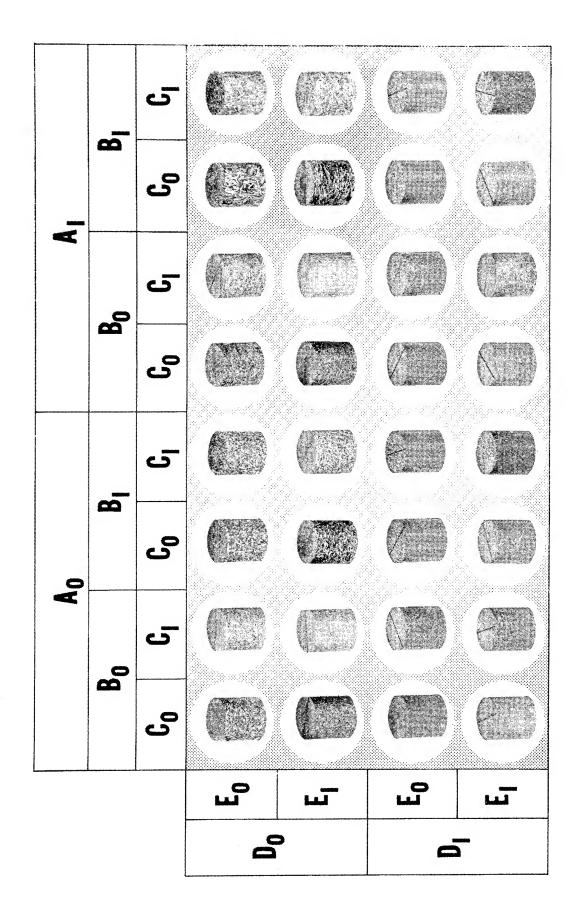


Figure 11. Factorial Design With Five Factors at Two Levels (3/4 Inch Sample).

The object is now to find the estimated main effects and interactions which also appear in a standard order:

T, A, B, AB

C, AC, BC, ABC etc...

where

T corresponds to the overall average effect

A corresponds to the main effect of factor A

AB corresponds to the interaction of factors A and B, etc.

d. Estimation of main effects and interactions. Yates' method is a systematic method for obtaining estimates of main effects and interaction for two-level factorials. The method was originally described by Yates<sup>25</sup> and may be found in various textbooks.<sup>26</sup>, <sup>27</sup>

The systematic procedure for Yates' method is as follows:

- (1) Make a table with n+2 columns. In the first column, list the treatment combinations in standard order.
- (2) In column 2, enter the observed response corresponding to each treatment combination listed in column 1.
- (3) In the top half of column 3, enter, in order, the sums of consecutive pairs of entries in column 2. In the bottom half of the column, enter, in order, the differences between the same consecutive pairs of entries, i.e., second entry minus first entry, fourth entry minus third entry, etc.
- (4) Obtain columns 4, 5 ..., n + 2 in the same manner as column 3; i.e., by obtaining in each case the sums and differences of the pairs in the preceding column in the manner described in step 3.

- (5) The entries in the last column (column n + 2) are called  $g_T$ ,  $g_A$ ,  $g_B$ ,  $g_{AB}$ , etc., corresponding to the ordered effects T, A, B, AB, etc. Estimates of main effects and interactions are obtained by dividing the appropriate g by  $2^{n-1}$ .  $g_T$  divided by  $2^{n-1}$  is the overall mean. Note: The remaining steps of this procedure are checks on the computation.
- (6) The sum of all the  $2^n$  individual responses (column 2) should equal the total given in the first entry of the **last** column (column n + 2).
- (7) The sum of the squares of the individual responses (column 2) should equal the sum of the squares of the entries in the last column (column n + 2) divided by  $2^n$ .
- (8) For any main effect, the entry in the last column (column n + 2) equals the sum of the responses in which that factor is at its higher level minus the sum of the responses in which that factor is at its lower level.

# e. Testing for significance of main effects and interactions.

- (1) Choose  $\alpha$  , the level of significance. (See Appendix B).
- (2) If there is no available estimate of the variance due to experimental error, find the sum of squares of the g's corresponding to interaction of three or more factors.
- (3) To obtain S<sup>2</sup>, divide the sum of the squares obtained in step 2 by  $2^n$   $\nu$ , where  $\nu$  is the number of interaction included. In a  $2^n$  factorial, the number of third and higher interactions will be

$$\nu = 2^{n} - \frac{1}{2} (n^{2} + n + 2)$$
 (80)

If an independent estimate of the variation due to experimental error is available, use this  $S^2$ .

- (4) Look up  $t_1$   $\alpha/2$  for  $\alpha$  degree of freedom. <sup>28</sup> If higher order interactions are used to obtain  $S^2$ ,  $\nu$  is the number of interactions included. If an independent estimate of  $S^2$  is used,  $\nu$  is the number of degrees of freedom associated with this estimate.
  - (5) Compute the range W:

$$W = (2^n)^{1/2} t_{1 - \alpha/2} S$$
 (81)

- (6) For any main effect or interaction X, if the absolute value of  $g_X$  is greater than W, conclude that X is different from zero; e.g., if  $|g_A| > W$ , conclude that the A effect is different from zero. Otherwise, there is no reason to believe that X is different from zero.
- (7) Yates' method for obtaining estimates of main effects and interaction for two-level factorials can be found in Appendix C.

# C. Specimen Fabrication and Processing

## 1. Introduction

The impact and widespread use of fiber reinforced plastics to date has been largely due to development of specialized fabrication techniques by the manufacturer. If components with certain specified characteristics, such as the specimens used in this study, are desired, the manufacturer does not know what to do, and their suggestions are quite expensive. It is, then, believed that in the future composite, fabrication will no longer be the sole province of the manufacturer, since it has become increasingly clear that the designer and fabricator must work together as part of an integrated effort if the full potential of fiber reinforced composites is to be realized for structural applications. Each must rely on the other for guidance. Future structural requirements will be too high to be met by the traditional empirical approach of the molding shop. In turn, optimization of design is meaningless if it results in a structure that cannot be produced.

#### 2. Materials

Each material has its own peculiar characteristics which determine its utility in composite and which dictate how the composite must be processed. For instance, screw plasticization of thermosets results in a frozen screw.

The properties of each component must be understood in order to capitalize on its desirable properties and avoid degrading the resulting composite through its misuses. The materials used in this study are:

a. Thermosetting resins: epoxies. <sup>29</sup> Enlarged ten million times, the molecules of these resins might resemble short pieces of thread, varying in length from half an inch in some of the liquids to several inches in the solids. When cured or hardened, these threads are joined together at the ends and along the sides to form large, cross-linked structures. Each molecule is tied to several others, like the rope of a fish-net or the filaments of a spider's web, but in an irregular, rather than neat, pattern. If the final products are solids, they may become soft when heated, but will never again liquify. The thermosetting specimens used in this study are made of Epon 828 and Versamid 140 in the ratio of 60 and 40 respectively.

"Epon 828" is manufactured by the Shell Chemical Corporation; it is a liquid, aromatic epoxy in which "diepoxide 0" is the predominant constituent (the "0" indicates the absence of hydroxyls).

Amine type compounds are the most widely used of all curing agents for epoxies. They are fast curing in room temperature, chear, low in viscosity, readily miscible. When cured, epoxies tend to be brittle.

Toughness, the opposite of brittleness, depends upon both tensile strength and elongation. If the stress-strain behavior of the material is measured, then the stress at break is the tensile strength; the strain at break is the elongation; and the area under the curve, the work input, is the toughness. Unmodified epoxies, highly cross-linked with aromatic amines, are hard materials with high tensile strength but low elongation. At the other extreme, with moderate elongation but low tensile strength, are materials such as swiss cheese. Both have low areas under the stress-strain curve; both are brittle. Somewhere in between, combining strength and stretch, are tough compositions needed in many industrial applications.

Different approaches have been used to provide more stretch without lessening the tensile strength. The "Versamid" resins have been used in this experiment as the curing agent for Epon 828. The "Versamid" resins are viscous, brown liquids developed commercially by General Mills, Inc. The following advantages are found when used in epoxy formulations:

- (1) Wide range of compatability with epoxy resins.
- (2) Long working life.
- (3) Convenient handling without need for solvents.
- (4) No need for volatile, toxic, explosive, or temperature sensitive curing or accelerating agents.
- (5) Low reaction temperature, therefore, they can be cast in large volumes.
- (6) Low shrinkage on cure and exceptional dimensional stability.
  - (7) Excellent resistance to mechanical impact or shock.
  - (8) High flexural, compressive, and tensile strength.
  - (9) Exceptional resistance to thermal shock.
  - (10) Good electrical resistance.
- (11) Excellent adhesion and bonding to a wide variety of materials.
  - (12) Good machineability.
- (13) Possibility for a wide range of properties from hard and strong at one extreme, to soft and resilient on the other.
- b. Epoxy titanium powder. This combination was chosen because of the excellent adhesion and bonding properties of the epoxy resin (Epon 828 Versamid 140) to the powder and the fibers.

c. Reinforcing agent. The reinforcing agent is, as the term implies, the primary load or heat carrying component. Consequently, for both efficient performance and cost effectiveness, it must be selected in terms of type, form, and quantity to meet the specific need.

In this study, discontinuous copper and aluminum fibers were used to increase the thermal conductivity of the matrix resins.

# 3. Manufacturing Setup and Fabrication Techniques

Fiberglass reinforced plastics (FRP) are, by far, the best developed and understood of man-made fibrous composites. More types of composites are prepared and more resins used with glass fibers than with any other reinforcing agent. Consequently, review of the fiberglass reinforced plastics technology serves to summarize the state-of-the-art for composite fabrication and processing.

The principal FRP fabrication methods can be conveniently divided into open mold and closed mold processes. Generally speaking, the open mold techniques can be characterized as yielding larger, higher performance composites, requiring more labor but less investment, having a slower production rate, and being highly dependent for quality on operator skill.

The closed mold processes, on the other hand, yield more reproducible composites but require much higher investment.

In this study, a closed mold process had to be used to obtain specimens specified by the factorial experiment. Figures 12, 13, and 14 show the ingredients and setups for manufacturing the specimens. The main design consideration is that of a smooth surface so that the parts can be easily released from the mold. It is also essential that the molds

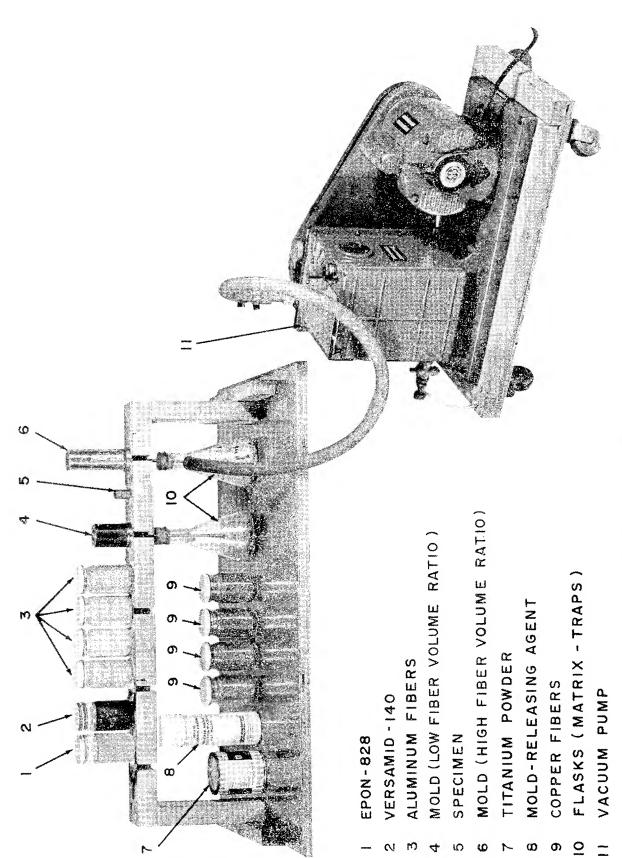


Figure 12. Vacuum Casting Setup.

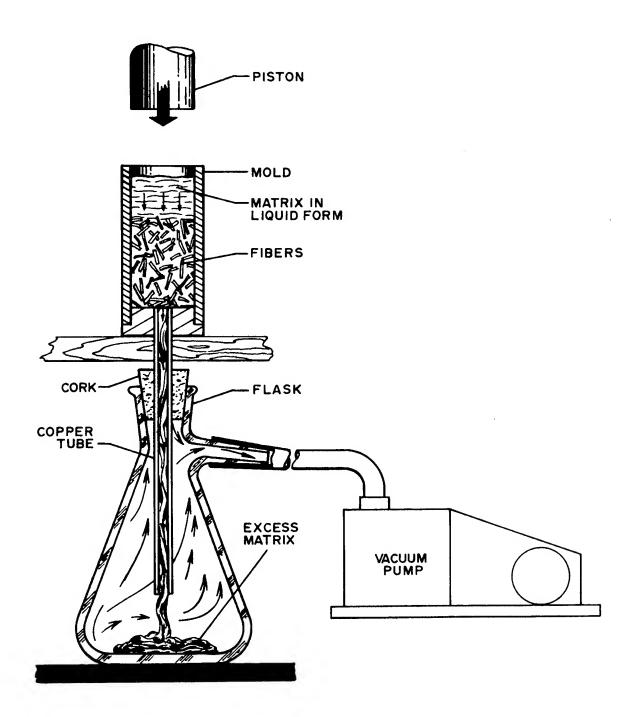


Figure 13. Cutaway View of the Vacuum Casting Process.

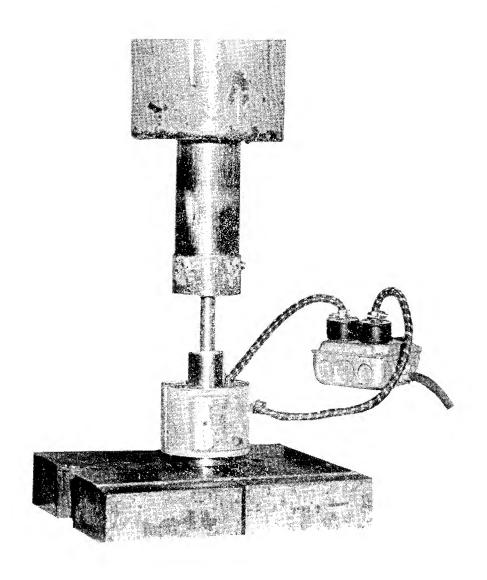


Figure 14. Setup for High Fiber Volume Ratio.

be rigid so that the dimensions of the part can be controlled. In this study, the molds were made of forged tool steel, hardened and polished. The vacuum casting technique with hot-pressing was used in the process of the specimens (Figures 12, 13, and 14). The main advantage of this process is the ability to make specimens free of voids.

The following steps should be taken to manufacture experimental specimens:

- a. Decide on the size of the mold and obtain the total volume  $(V_{\mathbf{T}})$  of the finished specimen.
- b. Weigh fibers, as specified by a predetermined volume ratio, per equation:

$$W_{f} = P_{f} V_{T} V_{R}$$
 (82)

where

Wf = Weight of fibers

 $\rho_{\rm f}$  = Density of fiber material

 $V_T$  = Total volume of finished specimen

 $V_{\rm p}$  = Fiber volume ratio

- c. Place fibers loosely in the mold.
- d. De-air matrix material by placing the proper amount of Epon 828 and Versamid 140 (in different cups) in a dessicator.
- e. Mix the portions of Epon 828 and Versamid 140 and de-air again.
  - f. Pour this matrix on the loose fibers.
  - g. Start vacuum pump to wet fibers.
- h. As soon as matrix shows in trap flask, stop pump, remove copper tubing from the mold, and set up the mold in a press.

- i. Place heating element around the mold, start pressing fibers to the predetermined  $V_{\mathrm{T}}$ , and leave in that position for twenty minutes.
  - Remove specimen from mold.
- If a check on the volume ratio is desired, weigh composite specimen and use the following equation for  $\boldsymbol{V}_{\boldsymbol{R}}\colon$

$$V_{R} = \frac{\frac{W_{c}}{V_{T}} - \rho_{m}}{\rho_{f} - \rho_{m}}$$
(83)

where

W = Weight of specimen

 $V_T$  = Total volume of specimen

 $\rho_{\rm f}$  = Density of fibers

 $\rho_{\rm m}$  = Density of matrix

In this study: (1) The finished specimen was cut into two samples, 1/4" and 3/4" respectively (Figure 15 and 16). (2) The volume ratio of Epon 828 and Versamid 140 is 60/40 respectively. (3) The volume ratio of Epon 828, Versamid 140, and Titanium powder is 48-32-20 respectively.

The following list shows some types of defects in the finished product and the possible causes:

Defect	

#### Possible Causes

sufficient pressure, dirty mold.

Blisters	Cure to rapid, mold too hot, moisture in resin or filler.
Pin holes, pits and voids	Poor mold surface, entrapped air, in-

Cure too rapid, mold too hot, insufficient Sink marks

pressure, poor port design.

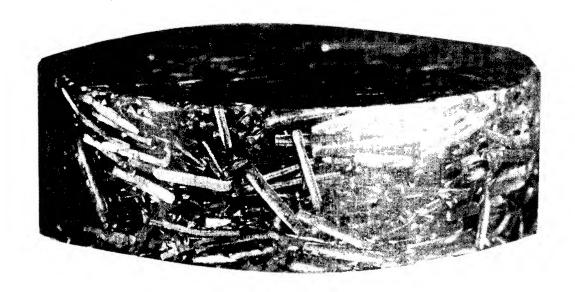


Figure 15. Enlarged View of a 1/4 Inch Specimen.

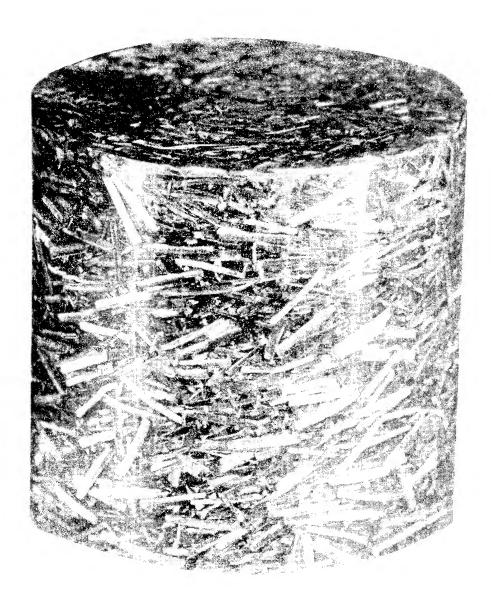


Figure 16. Enlarged View of a 3/4 Inch Specimen.

#### Defect

### Possible Causes

Crazing

Resin-rich areas caused by poor fiber

distribution.

Exposed Fibers

Fibers content too high, poor fiber distribution, too much mold lubricant, mold too hot.

Color variation

Poor fiber distribution.

Delamination

Poor impregnation, fiber content too

high.

#### EXPERIMENTAL PROCEDURE

# Apparari Used for Determining the Thermal Conductivity of Composite Specimens

# 1. Colore Thermoconductometer 50

a. Principle. This instrument, Figure 17, was developed by Dr. Johann Schroeder at Philips Zentraliaboratorium, Aachen, Germany. In lower vessel 11, a pure liquid is heared to its boiling point. The vapor impinges onto a silver plate 12 and then condenses in condenser 6 from which the cordencate returns through the overflow into vessel 11. Thus, the temperature of silver plate 12 can be maintained at the boiling temperature of liquid A. If the temperature of the silver plate drops even slightly below this boiling point, vapor condenses on the plate and transfers thereto its heat of condensation, which is more than twice that of the heat capacity of the same amount of liquid.

An upper vessel 7 contains another pure liquid B, the boiling point of which is about  $10^\circ$  -  $20^\circ$ C lower than that of the liquid in vessel 11. The bottom of vessel 7 is formed by another silver plate 13.

A cylindrical test sample S is placed between the two plates. The heat flowing through the sample from 12 to 13 causes liquid B to boil, thereby maintaining the temperature of the upper plate at the boiling point of the upper liquid. Thus, there is a constant temperature difference  $(T_A-T_B)$  between the two plates. The vapor from the upper liquid is condensed in condenser 8 and the condensate is collected in burette 14. When the condensate reaches the 0-mark 10 on the burette, a steady state

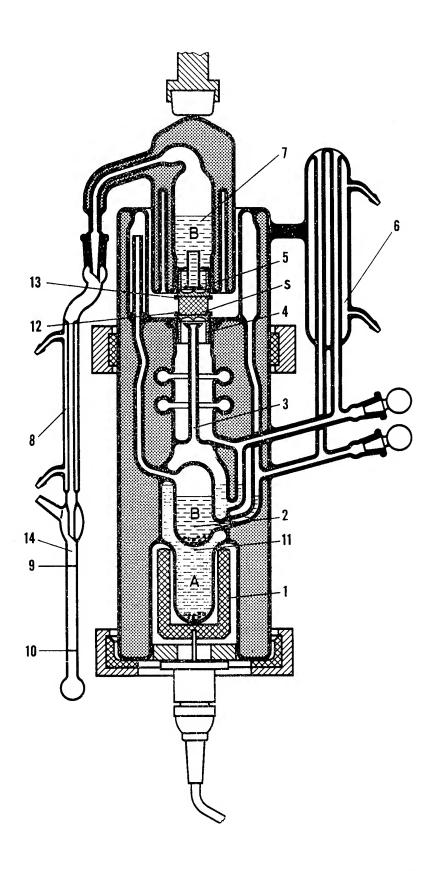


Figure 17. Schematic of Colora Thermoconductometer.

will have been reached. The time taken for distilling 1 ml of liquid to 0-mark 9 is determined with a stopwatch. The thermal conductivity  $K_{\rm B}$  is then given by

$$K_{B} = \frac{Q}{t \cdot (T_{A} - T_{B})} \cdot \frac{L}{A}$$

$$= \frac{L}{R \cdot A} \quad \text{cal/cm sec }^{\circ}C$$
(84)

where

Q = Heat of vaporization for 1 ml of liquid B

t = Time in seconds for distilling 1 ml

T<sub>A</sub> - T<sub>B</sub> = Temperature difference in °C which is given by the boiling points of the two liquids

L = Sample height in cm

A = Sample cross-section in  $cm^2$ 

R = Heat resistance of sample sec ° C/cal

In this way, a determination of the absolute thermal conductivity can be made in a few minutes by a simple time measurement. When a number of calibrated samples are available,  $K_{\mbox{\footnotesize{B}}}$  can be determined more easily by a comparative measurement without knowing the exact values of the boiling point and the heat of vaporization of liquid B.

b. Construction of the apparatus. A higher boiling liquid A is boiled by an electric heater 1 at the bottom of the glass apparatus. The vapor passes upward through pipe 3, impinges on stopper 4, flows to condenser 6, and returns as a liquid to the same place where it started. In the upper part of the apparatus 7, liquid B, at a boiling point 10-20°C lower than liquid A, is boiled by heat from stopper 5 which has passed to it through sample S. Vapor of B passes to condenser 8 and is collected in graduated cylinder 9-10.

To minimize heat loss from the upper vessel containing liquid B, a small boiler 2 of the same liquid B is operated in the liquid A, the vapor of which heats a jacket around the B liquid at the top and is condensed in condenser 6, and then returns to the bottom at boiler 2.

c. Preparation and measurement. Liquid pairs can easily be found for the whole measuring range from 20-200°C, having suitable boiling points differing by 10-20°C. In Table I, some pairs are summarized with their boiling points and the corresponding measuring temperatures  $T_M$ , i.e., the average temperature 1/2 ( $T_A + T_B$ ). In this connection, it must be remembered that ether and carbon disulphide are easily flammable and combustible. It is important that the liquids be pure and have a definite boiling point.

The heating power of the element has to be high enough so that liquid A boils steadily in order to get a constant heat transfer to the sample. To determine correct power inputs of the heating element for a particular liquid pair, the times are taken which are necessary to test a sample for different power inputs. In Figure 18, the measuring time, t, of the liquid pair, carbon disulphide and ether, is plotted against the electric power input, W. Curve II, in which the measuring time is independent of the power input, indicates the correct heating power. In curve I, the heat supply is too small, while in curve III, the boiling point is elevated because of the excessively high vapor pressure which consequently increases the temperature difference, thus decreasing the measuring time. In Table II, some selected values of the heating power are given for several liquid pairs.

The samples, used both for calibration and measurement, are

TABLE I

Liquid A	Liquid B	T <sub>A</sub> °C	T <sub>B</sub> °C	T <sub>M</sub> °C
Methylene Chloride	Freon 11	41.6	23.8	32.7
Carbon Disulphide	Ether	46.3	34.6	40.5
Water	Trichloroethylene	100.0	87.0	93.5
Monochlorobenzene	Toluene	132.0	110.8	121.4
o-Dichlorobenzene	Monobromobenzene	179.0	155.6	167.3

TABLE II
SELECTED VALUES OF THE HEATING POWER

Liquid Pairs	Heating Power		
Freon 11/Methylene Chloride	4.75		
Ether/Carbon Disulphide	4.5		
Trichloroethylene/Water	6.0		
Toluene/Monochlorobenzene	6.5		
Monobromobenzene/o-Dichlorobenzene	7.5		

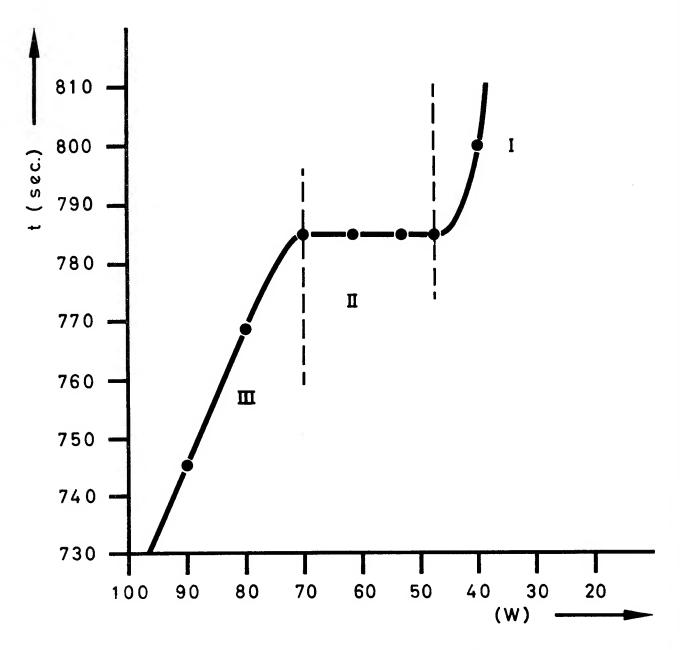


Figure 18. Plot of the Electric Power Output W of Heating H Against the Measured

Time t for the Liquid Pair, Carbon Disulphide/Ether.

cylindrical in form and can range in diameter from 16-18 mm and in length from 1-30 mm, depending on the thermal conductivity of the material. Samples of materials having a high thermal conductivity (pure metals) can be made in the form of hollow cylinders with a wall thickness of about 2 mm.

To minimize the contact resistance between sample and silver plates, it is very important that the cylinder-end surfaces of the sample be carefully ground flat and have as good a finish as can be obtained.

Ideally, the surfaces of the sample should be near parallel.

The remarkably good accuracy of measurement is due to the fact that the heat losses between the lower silver plate 13 and the upper liquid B are kept relatively low. This is achieved primarily by choosing the dimension of the sample so as to ensure a reasonably large heat flow through the sample. For instance, if the substance is a poor conductor of heat, the sample taken should be in the form of a thin disk. For a better conductor, a thicker sample or even a hollow cylinder should be used. However, an excessively large heat flow will cause liquid B to boil too violently and thus decrease the accuracy of the measurement. The height and cross section of the cylindrical sample should therefore be adapted to the estimated order of its thermal conductivity. The measuring time should not be less than 80 seconds and not more than 1000 seconds, while for the most exacting measurements, the time should be between 100 and 500 seconds (Figure 19).

A calibration curve has to be obtained for each liquid pair used. Because the curve is a straight line, it is usually sufficient to measure only two or three calibration samples. Figure 20 shows some of

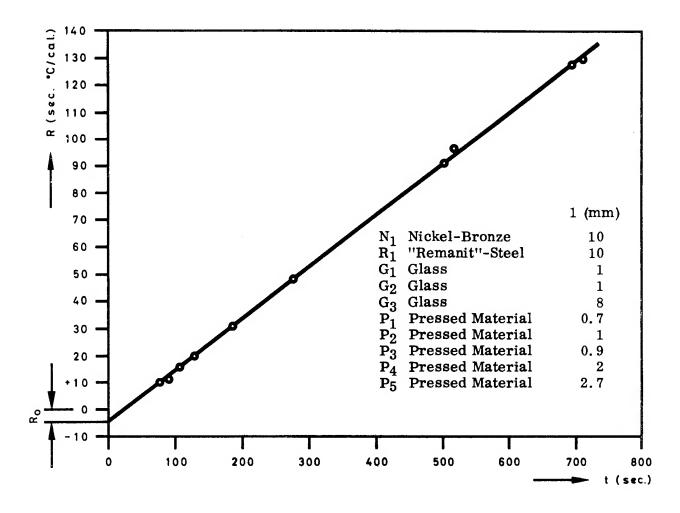


Figure 19. Calibration Diagram for an Apparatus Using the Liquid Pair, Carbon Disulphide/Ether as Boiling Liquids. The Known Thermal Resistance R of Ten Calibrated Samples is Plotted Against the Measured Time t Taken for 1 ml of Condensate to be Collected in Burette 14.

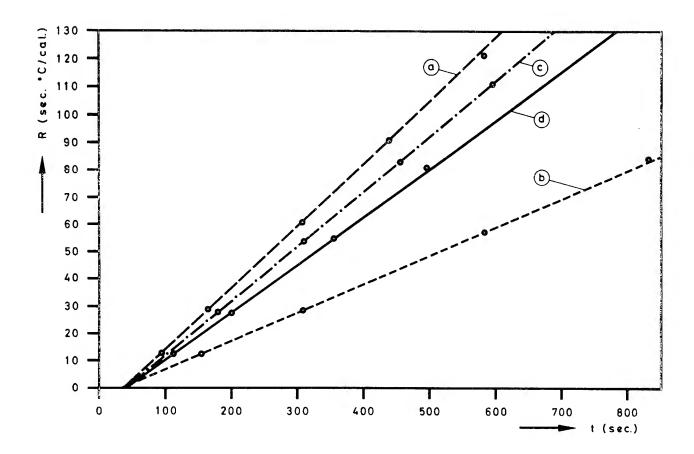


Figure 20. Calibration Diagram for the Following Liquid Pairs: a. Freon 11 (b.p. = 23.8°C) and Methylene Chloride (b.p. = 41.6°C). b. Trichloroethylene (b.p. = 87°C) and Water (b.p. = 100°C). c. Toluene (b.p. ==110.8°C) and Monochlorobenzene (b.p. = 132°C). d. Monobromobenzene (b.p. = 155.6°C) and o-Dichlorobenzene (b.p. = 179°C).

the calibration curves for several liquid pairs. They have to be measured anew for each apparatus. The diagrams of Figures 17 and 18 are used <a href="here">here</a> only as examples.

It should be noted that for these relative measurements it is not necessary to know the heat of vaporization or the exact values of the boiling temperatures. This at once eliminates the errors that can be introduced into a calculated value of  $K_{\mathrm{R}}$  if the boiling points of the liquids should differ slightly from the assumed values, owing to impurities, or if the ends of the samples should not attain the exact temperatures  $\boldsymbol{T}_{\boldsymbol{A}}$  and  $\boldsymbol{T}_{\boldsymbol{B}}\text{, owing to contact resistances.}$  In order to obtain reproducible results, care must be taken to ensure that these resistances and also the magnitude of the small, residual heat losses remain constant for all measurements. Because of the protective envelopes, the residual heat losses are negligible in comparison with the great amount of heat conducted through the sample. Using the contact oil (graphite filled oil) and carefully ground samples, the contact resistances are reduced and kept constant to such an extent that their effect in all measurements will normally remain below the limits of accuracy determined by other factors.

# 2. Comparative Thermal Conductivity Measuring System 31, 32, 33, 34, 35

a. <u>Principle</u>. The Comparative Thermal Conductivity Instrument, Figure 21, is used for determining the thermal conductivity of solid materials. In principle, the ability of the sample material to transport heat is compared to the ability of a known reference material to transport the same amount of heat.

The ability to transport heat through conduction is usually determined by measuring the temperature difference between two points in a



Figure 21. Comparative Thermal Conductivity Apparatus.

material when heat flows from one point in the direction of the other. The temperature difference ( $\Delta$ T) is related to the amount of heat flow (Q) by Fourier's heat conduction equation:

$$\Delta T = \frac{\Delta x}{K} \left(\frac{Q}{A}\right) \tag{85}$$

where  $\Delta$  x is the distance between the two temperature measuring points, A is the area of the cross section perpendicular to the direction of heat flow, and K is the coefficient of thermal conductivity.

It is quite obvious from the above equation that when the quantity Q/A is kept the same for two materials, one of known thermal conductivity (K reference) and one of unknown thermal conductivity ( $K_{\rm R}$  sample), the respective temperature drops are related as follows:

$$\left(\Delta T \frac{K_B}{\Delta x}\right)$$
 sample =  $\left(\Delta T \frac{K}{\Delta x}\right)$  reference

By measuring the temperature difference between two thermocouples a distance  $\Delta x$  apart in both reference material and test sample, the unknown thermal conductivity is

$$K_{B} = \left(K \frac{\Delta T}{\Delta x}\right) \text{ reference } \left(\frac{\Delta x}{\Delta T}\right) \text{ sample}$$
 (86)

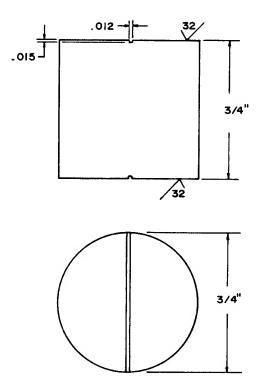
The condition of equal heat flux (or heat flow density, Q/A) is obtained by placing a test sample and reference material of equal cross section in intimate contact with each other and then by holding the two between a heater and a heat sink.

In practice, a number of precautions must be taken, however, to ensure a constant and uniform heat flux in the sample and reference material. First, heat losses (or gains) along the sides will occur whenever the temperature of the surrounding insulation does not exactly match those along the test stack. The fact that a temperature gradient exists along the test stack from the heater to the heat sink poses a difficult guarding problem. Second, even if the total amount of heat flow (Q) were kept constant, it would still be possible to obtain a non-uniform heat flux between the thermocouples, for example, if the disturbances caused by an irregular interface contact have not subsided at the location of the thermocouples.

To avoid these problems, it is necessary always to have the surfaces of the sample and reference materials prepared to as good a surface finish as can be attained under the circumstances. Radial heat transfer is reduced by placing the test stack inside a cylindrical furnace with a linear temperature gradient along the length of the tube. The end temperatures match those at adjacent points in the stack. The space between the furnace tube and the stack is filled with insulating powder.

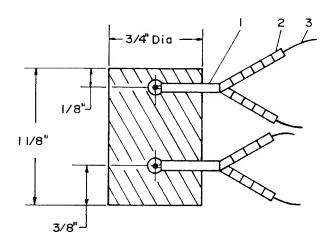
To obtain the best accuracy, the sample should be sandwiched between two identical reference materials having a thermal conductivity of the same order as that expected for the test material. The thermal conductivity is now determined as follows:

$$K_{B} = \frac{1}{2} \left( \frac{\Delta x}{\Delta T} \right) \text{ sample } \left[ \left( K \frac{\Delta T}{\Delta x} \right)_{\substack{\text{top ref-} \\ \text{erence}}} + \left( K \frac{\Delta T}{\Delta x} \right)_{\substack{\text{bottom} \\ \text{reference}}} \right]$$
(87)



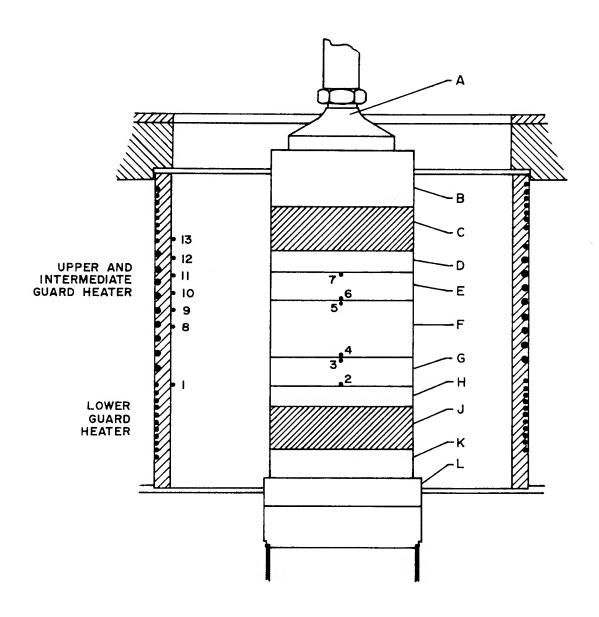
NOTE: TOP AND BOTTOM SURFACES FLAT AND PARALLEL TO 0.002"

Figure 22. Suggested Sample Geometry for Composite Specimens.



- I DOUBLE-BORE ALUMINA THERMOCOUPLE TUBING
- 2 ALUMINA BALL AND SOCKET BEADS
- 3 CHROMEL-ALUMEL BARE THERMO-COUPLE WIRE

Figure 23. Instrumentation for Pyrex and Pyroceram Reference Standards.



A = Pressure Pad

B = Insulation

C = Main Heater

D = Surface Plate

E = Top Reference Standard

F = Test Sample

G = Bottom Reference Standard

H = Surface Plate

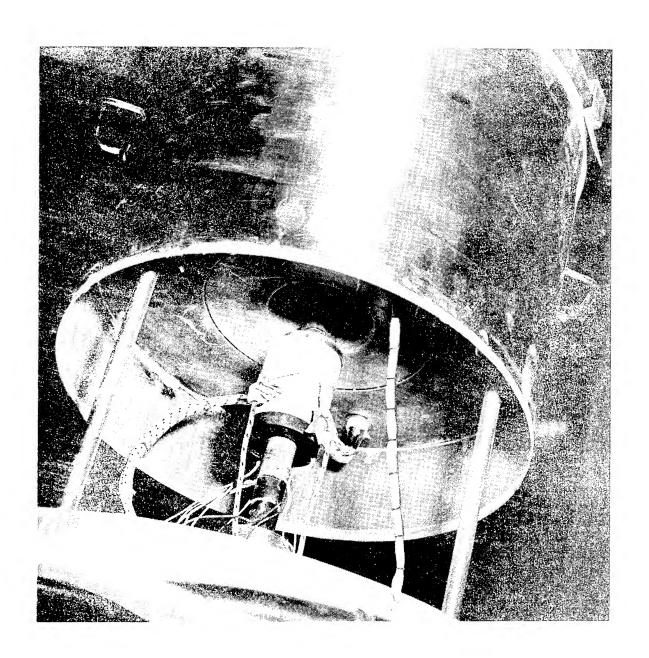
J = Auxiliary Heater

K = Insulation

L = Heat Sink

1-13 = Thermocouples

Figure 24. Schematic of Test Stack.



igure 25. Test Stack of Comparative Thermal Conductivity Apparatus.

The thermal conductivity of the reference materials is taken at the average temperature of each. In general, there are four materials which are used with the apparatus. These are, in the order of increasing thermal conductivity, Pyrex 7740, Pyroceram 9606, Inconel 702, and Armco iron. It is always desirable to have the temperature difference across the sample comparable to that across the reference materials, and thus the geometrical factor should be considered in relation to the choice of standards. Whereas the temperature gradient through a given test sample is determined by the output from the main heater only, the desired temperature level of the sample is obtained by adjusting the power into an auxiliary heater placed between the lower reference material and the heat sink.

b. Operation and construction. After the reference materials and the test specimen have been instrumented according to the instructions given in Figures 22 and 23, the test stack can be built as shown in Figure 24 or Figure 25. Thermocouples are connected to the terminal strips on the base plate following the numbering scheme from Figure 24. Attention must be paid to the fact that the correct height above the heat sink is obtained before the lower reference standard is put in place. This is to ensure that thermocouples No. 1 and 2 line up properly. Therefore, an aluminum block is supplied to replace the auxiliary heater (J) plus insulation (K) for tests at the very lowest temperatures. A similar piece of aluminum is supplied to replace just the insulation (K) for tests at moderately low temperatures. When the furnace is lowered, the space surrounding the stack is filled with insulating powder, such as bubbled alumina. Finally, the bell jar is placed over the test section and the test can begin. For other details, see reference 36.

# c. Test procedure.

- (1) Build the test stack according to instructions given in preceding paragraph 2a.
  - (2) Lower the guard furnace.
- (3) Fasten the coolant connections in the back of the furnace.
- (4) Fill the space inside the furnace tube with bubbled alumina or with some other suitable insulation.
  - (5) Place the bell jar over the test section.
- (6) Check continuity of all thermocouples using the potentiometer or a simple ohmmeter connected across the potentiometer outlet on the front panel.
- (7) Turn on coolant water to the instrument and verify that an adequate flow passes through. (For tests using liquid nitrogen as the coolant, regulate the flow by hand so that only a minute amount of liquid nitrogen flows through the outlet tube. After a brief period of time a steady state will have been reached and no further flow regulation is necessary).
  - (8) Turn all four auto transformers to zero.
  - (9) Turn on the Main Power switch.
- (10) Set the red pointer of the OTC (Oven Temperature Control) at the desired maximum temperature and push the reset button.
- (11) Put the Temperature Reference toggle switch in the Temperature Reference position (this is up).
- (12) If it is desired to test in a vacuum, close the Back-fill valve and switch on the mechanical vacuum pump.

- (13) If a vacuum gauge is supplied with the instrument, turn on Vacuum switch. After a pressure of about 30 microns has been reached, the test can begin.
- (14) If it is desired to backfill the test section with argon or nitrogen, open the Backfill valve <u>first</u> and <u>then</u> turn off the vacuum pump.
- (15) If an automatic ice reference system is supplied with the instrument, turn on the Ice Reference switch.
- and the guard heaters. (The approximate settings of the auto transformers for obtaining a given test temperature level will become familiar to the operator after some experience has been gained with the instrument.)

  The reading (in millivolts) on the Temperature Reference meter allows a rapid evaluation of the main heater input, while the indicated temperature on the OTC may serve to provide a quick indication of the stack temperature level which is dependent on the auxiliary heater power. (This, of course, assumes that the OTC thermocouple has been placed in contact with, e.g., the main heater or test sample.) Power should be increased gradually at a rate at which the heater temperatures do not increase by more than about 300°F per hour. If one or two thermocouples are monitored on the potentiometer and the results plotted, the final equilibrium temperature may be roughly predicted and the power adjusted if it will be other than that desired.
- (17) Adjust the Lower Guard Heater to obtain TC 1 equal to TC 2.

- (18) Adjust the Upper Guard Heater to obtain the selected control thermocouple (see preceding paragraph 2b) reading equal to TC 7.
- (19) Wait for thermal equilibrium conditions to prevail throughout the test section. A sufficiently steady state has been reached when the temperatures do not change by more than about 1°F per half hour.
- (20) Record the temperatures along the test stack. Note
  1: the Ice Reference light must be cycling on and off. Note 2: the
  Temperature Reference switch must be set on TC Select before reading
  TC 4 and TC 5 on the potentiometer.
- (21) When the test is over, the vacuum may be broken by first opening the Backfill valve and then turning off the vacuum pump. If this order were reversed, the possibility exists that oil would be sucked from the pump into the chamber.
- d. Data reduction. In the preceding paragraph a, it was shown that the thermal conductivity of the sample ( $K_S$ ) is calculated from

$$K_{s} = \frac{1}{2} \left( \frac{\Delta x}{\Delta T} \right)_{s} \left[ \left( K \frac{\Delta T}{\Delta x} \right)_{tr} + \left( K \frac{\Delta T}{\Delta x} \right)_{br} \right]$$

where the subscripts

s = sample

tr = top reference standard

br = bottom reference standard

This equation may be rewritten as follows:

$$K_{s} = \frac{1}{2} \Delta x_{s} \left[ \left( \frac{K}{\Delta x} \right)_{tr} \left( \frac{\Delta T_{tr}}{\Delta T_{s}} \right) + \left( \frac{K}{\Delta x} \right)_{br} \left( \frac{\Delta T_{br}}{\Delta T_{s}} \right) \right]$$
(88)

The advantage is that the temperature differences may be substituted by millivolt differences obtained directly from the potentiometer readings. Generally, this method gives more accurate results, but it should be kept in mind that the scheme is admissable only as long as the total temperature drop through the stack is sufficiently small that the emf versus temperature relationship of a chromel-alumel thermocouple may be considered linear over this range. This is generally the case, and the errors in converting from emf to °F exceed those resulting from a possible, slight non-linearity.

Thus, we obtain:

$$K_{s} = \frac{1}{2} \Delta x_{s} \left[ \left( \frac{K}{\Delta x} \right)_{tr} \left( \frac{\Delta emf}{\Delta emf}_{s} \right) + \left( \frac{K}{\Delta x} \right)_{br} \left( \frac{\Delta emf}{\Delta emf}_{s} \right) \right]$$
(89)

where

 $\Delta x_s$  = distance between thermocouple junctions in the sample in ft.

 $\Delta x_{tr}$  = distance between thermocouple junctions in the top reference in ft.

 $\Delta x_{\text{br}}$  = distance between thermocouple junctions in the bottom reference in ft.

 $K_s$  = sample thermal conductivity in Btu/hr-ft degF evaluated at a mean temperature  $\left(\frac{TC4 + TC5}{2}\right)$  °F

 $K_{tr}$  = thermal conductivity of the top reference in Btu/hr-ft degF evaluated at a mean temperature  $\left(\frac{TC \ 6 + TC \ 7}{2}\right)$  °F

 $K_{br}$  = thermal conductivity of the bottom reference in Btu/hr-ft degF evaluated at a mean temperature  $\left(\frac{TC \ 2 + TC \ 3}{2}\right) \circ F$ 

 $K_{\mbox{tr}}$  and  $K_{\mbox{br}}$  are obtained from the calibration curves supplied with the apparatus.

$$\triangle \text{ emf}_{s} = (\text{TC 5 - TC 4}) \text{ mv}$$

$$\triangle \text{ emf}_{tr} = (TC 7 - TC 6) \text{ mv}$$

$$\Delta \text{ emf}_{br} = (TC \ 3 - TC \ 2) \text{ mv}$$

As a check on the accuracy of the test, one should verify that the total energy through the top and through the bottom reference standard is the same, or

$$K\left(\frac{\Delta T}{\Delta x}\right)_{tr} = K\left(\frac{\Delta T}{\Delta x}\right)_{br}$$

#### PART IV

#### DISCUSSION AND PRESENTATION OF RESULTS

# A. Materials 37, 38, 39

The observed general characteristics of the composite specimens are affected by many factors, the most important of which are:

- a. Properties of the resin-hardener-additive (Ti) formulation.
- b. Properties of the fibers.
- c. Quality of the resin to fiber bond.
- d. Proportion of fiber to resin.
- e. Orientation and distribution of fibers.

Some important aspects of performance expected from these composites are:

- a. Resistance to surface attack by water, chemicals, and solvents.
  - b. Long-term maintenance of mechanical and thermal properties.
  - c. Impermeability to liquids and gases.

Each of the aforementioned parts could be developed in an extensive research program. A few important observations are presented to facilitate the understanding of the experimental results. Appendix B comprises most of the definitions of the chemical terms used in this discussion.

As mentioned in Part II, the hardener used with Epon 828 was Versamid 140. This resin, made from polymerized vegetable oil acids or polyamid resins, is usually preferred since it has the lowest viscosity and highest heat distortion temperature obtainable with any Versamid. It has excellent adhesion to various substrates, outstanding impact resistance,

chemical and solvent resistance, low shrinkage, and low exotherm. The Versamid 140 also has free primary and secondary amine groups in the polyamide, along with some carboxyl and a mixture of condensation products. When mixed with epoxy resin, the amines, carboxyl, etc., are then available for curing.

Copper is one of the metals which reacts with amines, and the danger of both general corrosion and stress corrosion, if components are under stress, are present. The copper fibers used in this study were made from an electrolytic, tough-pitch copper sheet, the purest grade of copper commercially available. The minimum copper content of this material is 99.95 per cent, and the method of manufacture is such that no residual deoxidant is present (it contains only 0.04% oxygen). Because of these characteristics, the danger of corrosion of copper fibers, when mixed with the epoxy resins, is reduced.

The aluminum fibers were made from a 99.99% commercially available aluminum sheet. These fibers are highly resistant to most atmospheres and to a great deal of chemical agents. This resistance is due to the inert and protective character of the aluminum oxide film which forms on the metal surface. The amines are generally corrosive to aluminum, but 99.99% Al is, however, resistant to amines, unless they have a very high content of ammonia and ammonium hydroxide.

The Titanium powder (97.1 Ti; 0.02 Si; 0.70 N; 0.05 Fe; 0.09 H; 0.30 Ca; 0.30 Al; 0.01 Mg; 7.6 microns is the average particle size, having a 99.2 - 325 mesh) is intrinsically very reactive, so that whenever the metal surface is exposed to air, or to any environment containing available oxygen, a thin, tenacious surface film of oxide is formed; this oxide film

confers upon Titanium excellent corrosion resistance in a wide range of corrosive media, and it behaves very favorably in contact with other metals.

# B. Experimental Results

The experimental results, obtained from the "Colora" and "Comparative Apparatus", for the factorial experiments are presented in Tables III through V and conveniently plotted on a semi-log scale in Figures 26 thru 29. The results of Yates' factorial experiment, consisting of five factors at two levels, are presented in Tables VI and VII. Recall that the factors are the length of the fibre, the cross section of the fibre, the thermal conductivity of the fibre, the thermal conductivity of the matrix, and the fibre volume ratio. The analysis shows that the most meaningful factors to be considered for an optimum design of the type of composite in question are, in order of importance, the conductivity of the matrix, the fibre volume ratio, and the fibre cross section.

In general, the results show that the aluminum fibers have a greater effect on the composite thermal conductivity than the copper fibers although the thermal conductivity of copper is almost twice that of aluminum.

It would seem that there must be an added thermal resistance introduced between the copper fibers and the matrix. At first, it seemed that it could be due to a chemical reaction between the fibers and matrix, to an oxide film on the fibers, to the possibility that the matrix material does not adhere so well to copper as to aluminum, or a combination of the three.

A chemical reaction, after reviewing the properties of the materials involved, could have been possible. It is also reasonable to conclude that, if there was a reaction, the properties of the composite

TABLE III

DETAILS OF SAMPLES TESTED WITH THE "COLORA"

EPOXY MATRIX

EPOXY-Ti MATRIX

Sample	Diameter (mm)	Length (mm)	Weight (gm)	Density (gm cm <sup>-3</sup> )	Fibre Vol. Ratio	Sample	Diameter (mm)	Length (mm)	Weight (gm)	Density (gm cm <sup>-3</sup> )	Fibre Vol. Ratio
Matrix	19.04	2.44			1	Matrix	19.00	2.54	1.11	1.53	
	19.34	6.34	5.13	2.76	22.4	Q	19,34	6,39	6.03	3,23	22.8
А	19,10	6.24	4.75	2.66	21.0	AD	19,11	6.33	5,42	2.98	19,6
В	18,75	6.29	4.39	2.58	20.0	BD	18.92	6.34	5,39	3.02	20,1
AB	19,33	6.24	5,23	2,90	24.2	ABD	19,12	66.9	5,19	2.58	14.4
ш	19,20	6.35	7.70	4.19	41.6	DE	19.15	6.36	8.75	4.78	44.0
AE	19.35	6.32	7.54	4.07	40.0	ADE	19,17	6,35	7.30	4.00	33.5
BE	19,13	6.34	7.70	4.23	42.2	BDE	19.34	6.35	8.36	4.50	40.0
ABE	19.35	6.35	7.82	4.18	41.5	ABDE	19,35	6.34	8.14	4.35	38.2
ပ	19.08	6.36	2.64	1,45	20.8	CD	19,13	6.35	3,33	1.83	24.0
AC	19.08	6.32	2.59	1,43	19.7	ACD	19,09	6.36	3,14	1.73	16.0
BC	18.78	6.36	2.50	1.42	19.0	BCD	18,86	6,35	3.26	1.84	24.8
ABC	19.32	6.37	2.49	1,33	13.7	ABCD	19,01	6.36	3,16	1.75	17.6
CE	19,35	6.35	3,17	1.70	35.8	CDE	19,15	6.35	3,46	1.89	38.8
ACE	19,35	6.33	3.20	1.71	36.4	ACDE	19,08	6.36	3,88	2.15	49.6
BCE	19.20	6.35	3.20	1.74	38.1	BCDE	19,31	6.35	3,77	2.02	39.2
ABCE	19,34	6.37	3,28	1,75	38.7	ABCDE	19.36	6.34	3,61	1,94	32.8

TABLE IV

THERMAL CONDUCTIVITY MEASUREMENTS OBTAINED FROM THE "COLORA"

	7:1:1								
Sample	Fibre Vol. Ratio (%)	Time (Sec)	Range (Sec)	$(W \text{ m}^{*}\text{KB} \text{deg}^{-1})$	Sample	Fibre Vol. Ratio (%)	Time (Sec)	Range (Sec)	$(\text{W m}^{*K}\text{B}\text{deg}^{-1})$
Matrix		895	885-908	0.238	Matrix		594.0	592-597.8	0.365
7	22.4	223.4	222.5-224.4	2,63	D	22.8	144.3	143.9-145.0	4.53
¥	21.0	343.1	341.5-343.6	1,61	AD	19.6	203.7	203.4-203.9	2.98
ജ	20.0	528.6	525-533.2	1.04	BD	20.1	312.4	310-314.2	1.78
AB	24.2	263.3	258.5-265.9	2,13	ABD	14.4	276.2	273.8-280.5	2,29
ш	41.6	224.3	9-226	2.62	DE	44.0	81,5	80.2-82.1	10.0
AE	40.0	292.1	291,0-293,3	1.91	ADE	33.5	149.5		4.37
BE	•	364.4	359,5-369,6	1.52	BDE	40.0	177.2		3.46
ABE	41.5	307.6	302,7-312,0	1.81	ABDE	38.2	175.6	175,5-176,5	3,48
ပ	20.8	379.4	376-381.0	1.46	CD	24.0	203,4	206.6	
AC	19.7	335.6	329,9-340,1	1.67	ACD	16,0	214.9	209,8-219,6	2.82
BC	19.0	579.2	578.7-580	0.95	BCD	24.8	212.6		
ABC	13.7	295.0	292.8-297.6	1.91	ABCD	17.6	245.9	243-247	
CE	35.8	254.2	251,7-257,0	2.26	CDE	38,8	142.3	141-142.7	4.67
ACE	36.4	142.6	141-143.9	4.53	ACDE	49.6	71.8	71.5-72.4	12.05
BCE	38.1	284.6	283,7-286,0	2.02	BCDE	39,2	208,3	206.2-209.5	
ABCE	38.7	209.8	208.3-211.3	•	ABCDE	32.8	209,4	206,1-212,7	2.82
* *ABC	13.7	307.0	304,3-310,1	•	**ADE	33,5	155,7	153,7-159,6	•
**ACE	36.4	143.4	141.2-144.0	4.50	**ABCDE	32.8	206.1	~	2,85

 $<sup>^{*}</sup>K_{B}$  = Thermal Conductivity

<sup>=</sup> repeats

TABLE V

THERMAL CONDUCTIVITY MEASUREMENTS OBTAINED FROM THE "COMPARATIVE" METHOD

EPOXY MATRIX

EPOXY-Ti MATRIX

Sample	Density (gm cm <sup>-3</sup> )	Fibre Vol. Ratio (%)	$^{*}$ K $_{\mathrm{B}}$ (W m <sup>-1</sup> deg <sup>-1</sup> )	Temp. (°C)	Sample	Density (gm cm <sup>-3</sup> )	Fibre Vol. Ratio (%)	$^{*K}_{B}$ (W m <sup>-1</sup> deg <sup>-1</sup> )	Temp (°C)
Matrix	1.10		0.238	40.5	Matrix	1.53	1	0.365	40.5
1	2,80	21	2.66	53.0	D	2,96	19.7	2.66	53.1
A	2.66	20	1,64	48.8	AD	3.08	21,3	2,59	56.0
83	2.45	23	1,36	58.9	ВД	2.81	17.7	2.0	57.5
AB	2.92	28.8		63.0	ABD	2.98	19.8	2.58	56.7
ш	4.10	38.4	2,39	52.2	DE	4.42	39.4	6.13	55,2
AE	4.13	38.7	2.33	61.9	ADE	4.36	38.0	7.60	56.7
BE	4.28	40.7	1.76	58.1	RDE	4.61	42.0	5.18	64.5
ABE	4.16	39	1.60	57.5	ABDE	4.50	40.5	2.94	59.9
ပ	1.46	21.7		53.1	CD	1.90	28.2	2.61	53,7
AC	1.46	21	2,13	26.0	ACD	1.79	22.6	2,20	58,1
BC	1.33	22		54.5	BCD	1.81	24.2	3,46	58,4
ABC	1.47	21	2,10	57.1	ABCD	1.78	21.8	2,23	54.0
CE	1.74	38.4	4.61	58.2	CDE	1.94	31.2	3,26	53.0
ACE	1.76	39.6	3,56	55.2	ACDE	2.05	42.0	11,72	53,5
BCE	1.74	38.2	2,32	62.0	BCDE	2.06	43.7	2,80	0.09
ABCE	1.820	42.5	1.84	52.9	ABCDE	2.04	42.0	3,14	54.3

 ${}^*K_B$  = Thermal Conductivity

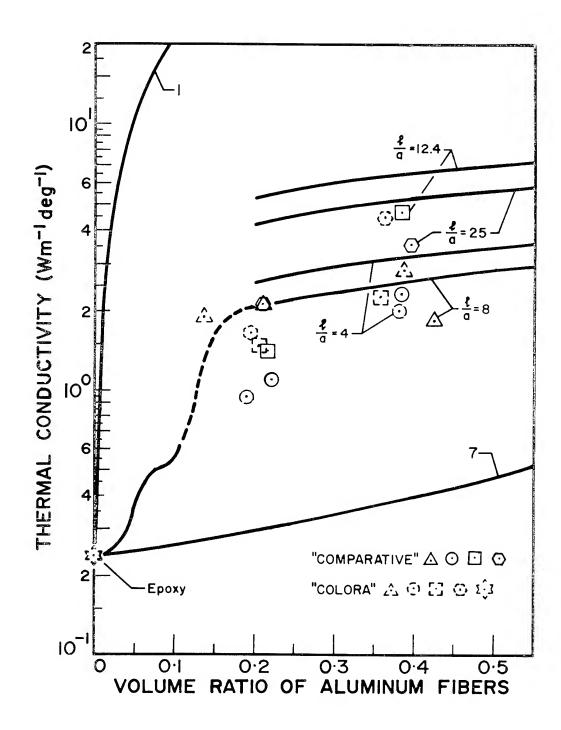


Figure 26. Experimental Values of Thermal Conductivity vs. Fiber Volume Ratio (A1-Ep). Curve 1 (Equations 2 and 22); Curve 7 (Equation 3).

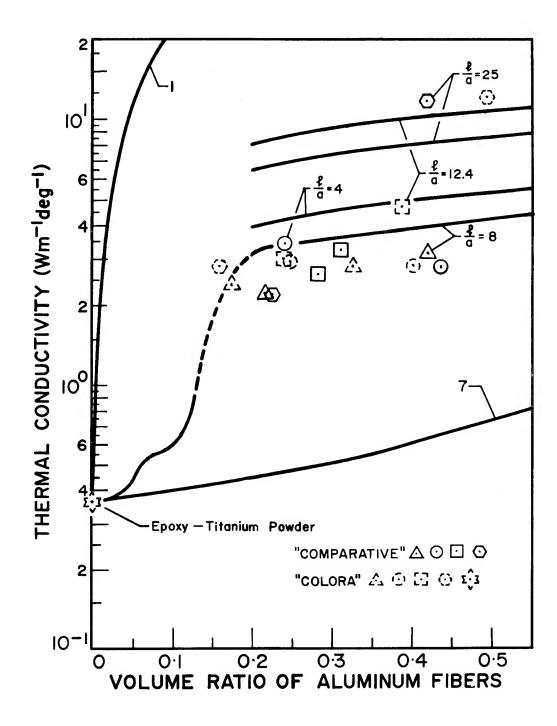


Figure 27. Experimental Values of Thermal Conductivity vs. Fiber Volume Ratio (A1-Ep-Ti). Curve 1 (Equations 2 and 22); Curve 7 (Equation 3).

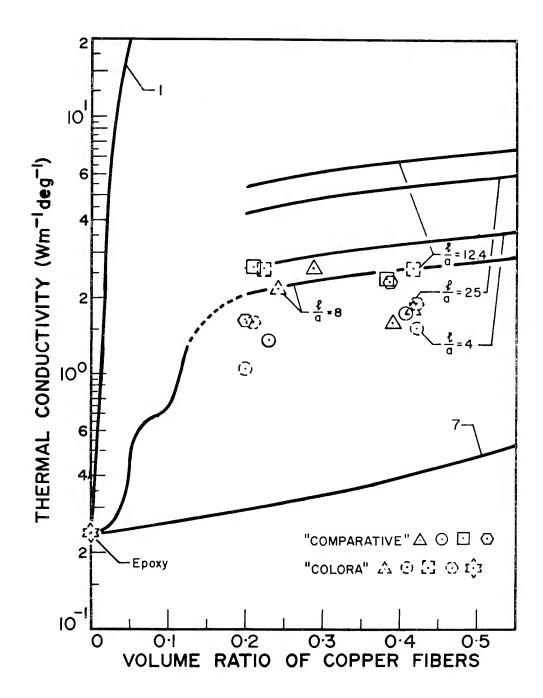


Figure 28. Experimental Values of Thermal Conductivity vs. Fiber Volume Ratio (Cu-Ep). Curve 1 (Equations 2 and 22); Curve 7 (Equation 3).

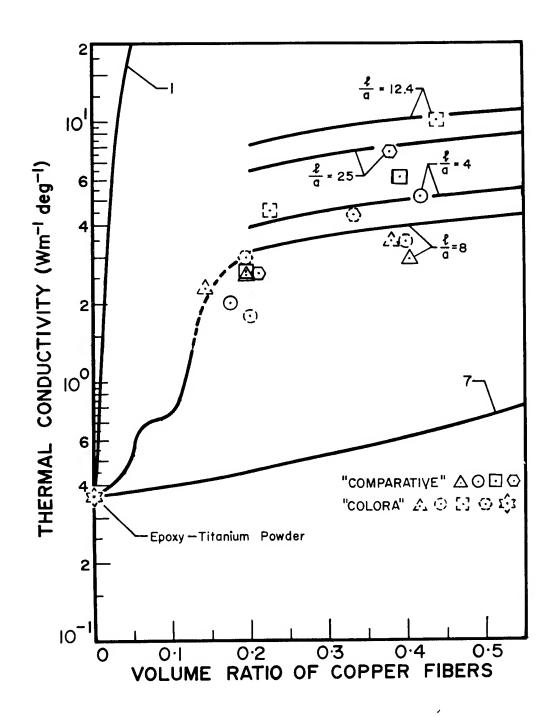


Figure 29. Experimental Values of Thermal Conductivity vs. Fiber Volume Ratio (Cu-Ep-Ti). Curve 1 (Equations 2 and 22); Curve 7 (Equation 3).

TABLE VI RESULTS FROM YATES' FACTORIAL EXPERIMENT ("COLORA")

TREATMENT COMBINATION	RESPONSE (KB)	:	ORDERED EFFECTS		ESTIMATED MAIN EFFECT
	(KB)  0.263000E 0.161000E 0.161000E 0.104000E 0.146000E 0.146000E 0.191000E 0.453000E 0.298000E 0.299000E 0.299000E 0.292000E 0.292000E 0.292000E 0.292000E 0.292000E 0.262000E 0.152000E	01 01 01 01 01 01 01 01 01 01 01 01 01 0	0.994600E 0.392000E -0.268600E 0.248000E 0.292000E 0.179400E -0.600000E -0.193400E -0.402000E -0.180400E -0.174000E -0.174000E 0.880000E -0.164600E -0.164600E -0.676000E 0.151000E -0.676000E 0.17400E 0.17400E 0.271000E 0.151000E -0.164600E -0.17400E 0.19600E -0.128000E -0.128000E -0.60000E	021021020000000000000000000000000000000	0.621625E 01 0.245000E 00 -0.167875E 01 0.155000E 00 0.182500E 00 0.112125E 01 -0.375000E-01 -0.120875E 01 0.209500E 01 -0.153750E 00 -0.112750E 01 -0.153750E 00 -0.169375E 01 -0.727500E 00 -0.412500E-01 -0.727500E 00 0.169375E 01 -0.422500E 00 0.422500E 00 0.422500E 00 0.943750E 00 -0.733750E 00 -0.733750E 00 -0.733750E 00 -0.80000E 00 -0.80000E 00 -0.80000E 00
CDE ACDE ABCDE ABCDE	0.468000E 0.120500E 0.285000E 0.282000E	01 02 01 01	-0.378000E 0.101600E -0.506000E -0.968000E	01 02 01 01	-0.236250E 00 0.635000E 00 -0.316250E 00 -0.605000E 00

SAMPLE	ESTIMAT	Ē	
OF VA	RIANCE	#	2.72713200

W# 19.80450

1	0.994600E	02
В	-0.268600E	02
D	0.335200E	02
Ε	0.271000E	02

TABLE VII

RESULTS FROM YATES' FACTORIAL EXPERIMENT ("COMPARATIVE")

TREATMENT COMBINATION	RESPONSE (KB)	Ξ	ORDERED EFFECTS		ESTIMATED MAIN EFFECT
1 A B A C A B A D A B A C A B A B A C A B A D A B A C A B A E A B A C A B A D A B A C	0.266000E 0.164000E 0.136000E 0.262000E 0.141000E 0.213000E 0.210000E 0.266000E 0.259000E 0.259000E 0.258000E 0.258000E 0.220000E 0.239000E 0.123000E 0.123000E 0.123000E 0.176000E 0.160000E 0.160000E 0.160000E 0.184000E 0.184000E 0.184000E 0.184000E 0.184000E 0.184000E 0.184000E 0.184000E	01 01 01 01 01 01 01 01 01	0.975300E 0.611000E -0.214700E -0.997000E 0.145000E 0.659000E -0.555000E -0.821000E 0.266700E 0.569000E -0.941000E -0.160300E -0.941000E -0.210000E -0.210000E -0.210000E -0.288300E 0.645000E -0.185700E -0.127500E 0.519000E 0.993000E 0.993000E -0.138700E 0.138700E -0.915000E -0.915000E -0.857000E -0.857000E -0.639000E	010010100100100000110000011000001101	0.609562E 01 0.381875E 00 -0.134187E 01 -0.623125E 00 0.906250E-01 0.411875E 00 -0.346875E 00 -0.513125E 00 0.166687E 01 0.355625E 00 -0.588125E 00 -0.588125E 00 -0.100187E 01 -0.248125E 00 0.390625E 00 -0.131250E-01 -0.346875E 00 0.180187E 01 0.403125E 00 -0.116062E 01 -0.796875E 00 0.324375E 00 0.456250E-01 0.413125E 00 -0.413125E 00 -0.456250E-01 0.866875E 00 -0.571875E 00 -0.535625E 00 -0.399375E 00
ACDE BCDE Abcde	0.117200E 0.280000E 0.314000E	02 01 01	0.155100E -0.135000E -0.461000E	02 01 01	0.969375E 00 -0.843750E-01 -0.288125E 00

SAMPLE ESTIMATE OF VARIANCE #	2.64390160	w# 19.49995
A 0753005 02		

1	0.975300E	02
В	-0.214700E	02
D	0.266700E	02
Ε	0.288300E	<b>J</b> 2

would not have changed enough to produce such high differences in thermal conductivities as in the case of sample ACE (Epoxy-Al fibers) which has a thermal conductivity almost two and a half times higher than its corresponding sample AE (Epoxy-Cu fibers).

The aluminum and copper fibers usually have a thin layer of oxide on their surfaces. Handbook values of thermal conductivities of copper oxide and aluminum oxide are 1.18 and 0.68 W m<sup>-1</sup> deg<sup>-1</sup> respectively. These values, when compared with the matrices conductivities of epoxy and epoxy-titanium (i.e., 0.238 and 0.365 W m<sup>-1</sup> deg<sup>-1</sup> respectively), are higher; hence the possibility of an additive resistance could be disregarded.

The bonding properties of the epoxy resin, as mentioned in Part

II, are excellent. Some air bubbles could have been trapped during the specimen fabrication; however, it is believed, after inspecting the specimens, that their effect on the composite thermal conductivity is negligible; i.e., very few air holes are visible for low volume ratios and none for high volume ratios.

The aluminum and copper filled specimens were made by the same method; hence, it was expected, at first, that the two sets of specimens had the same or similar fiber distribution. For the 20% fiber volume ratios, most of the fibers, when poured in the mold (Figure 13), filled the predetermined volume,  $V_{\rm T}$ , evenly, and there was no need to press them. For the 40% fiber volume ratios, and higher, the fibers had to be pressed down in order to fill  $V_{\rm T}$ . N.B. If two specimens have identical matrices, identical volume ratios, identical fiber geometry, but one has aluminum fibers and the other copper fibers, one would expect the copper filled

specimens to have higher thermal conductivity than the aluminum specimens, since the thermal conductivity of copper is about 80% higher than aluminum.

In the 20% fiber volume ratios, the aforementioned statement seems to apply. Tables IV and V, Figures 26 through 29, present the experimental thermal conductivities and, in all cases, the 20% fiber volume ratios show an increase in  $K_{\rm B}$  from the aluminum specimens to the copper ones, in the epoxy matrix as well as the epoxy-titanium powder matrix. Note, for example, the samples:

 $C \rightarrow K_B = 1.46$ , Al fibers (0.005 X 0.005 X 0.062),  $V_B = 20.8\%$ , Ep matrix

CD  $\rightarrow$  K<sub>B</sub> = 2.99, Al fibers (0.005 X 0.005 X 0.062), V<sub>R</sub> = 24%, Ep-Ti matrix

1  $\rightarrow$   $K_B$  = 2.63, Cu fibers (0.005 X 0.005 X 0.062),  $V_R$  = 22.4%, Ep matrix

D  $\rightarrow$  K<sub>B</sub> = 4.53, Cu fibers (0.005 X 0.005 X 0.062), V<sub>R</sub> = 22.3%, Ep-Ti matrix

The increase in  $K_{\rm R}$  from C to 1 = 80%

The increase in  $K_{\overline{B}}$  from CD to D = 51.5%

The increase in  $K_{\rm R}$  from C to CD = 105%

The increase in  $K_R$  from 1 to D = 74.5%

From the results, it is reasonable to assume that the 20% fiber volume ratio specimens, having the same fiber geometry, could have similar distributions.

In the 40% fiber volume ratios, half of the results show the aluminum filled specimens to be more conductive than the copper ones. This strange behavior could be explained if the manufacturing process and the mechanical properties of the fibers are considered. For this volume ratio, as previously indicated, the fibers had to be pressed. The copper fibers are stiffer than the aluminum ones; hence, when pressed, they tend to distribute themselves perpendicular to the applied pressure.

This behavior was examined in detail by fabricating a large specimen, 2-1/2 inches in diameter and three inches long (Figure 30). From this, four specimens were machined: two, 3/4 of an inch in diameter and 3/4 of an inch long, to be tested in the "Comparative Apparatus", and two, 3/4 of an inch in diameter and 1/4 of an inch long, to be tested in the "Colora". Note that the specimens were machined in the direction perpendicular to the Y axis. The four specimens have a fiber volume ratio of 40% and copper fibers (0.015 X 0.015 X 0.125) in an epoxy matrix. As was expected, their experimental results, shown in Table VIII, were much higher than their corresponding ABE samples in Tables IV and V. This clearly indicates that specimens having copper fibers can be fabricated with anisotropic characteristics by just pressing the fibers. Several private companies, universities, and governmental agencies, such as Wright-Patterson Air Force Base under the guidance of Dr. S. W. Tsai, and the University of Washington in St. Louis under the direction of Dr. Tolbert, are in the process of developing sophisticated methods (for instance, using a magnetic field) to align short fibers or whiskers; they feel that a successful but expensive process can be developed. What would happen when the fibers or whiskers are non-magnetic? It is believed that the simple process found in this study could be very useful in some applications. In reviewing the literature on composite materials, no one mentions this finding. The behavior of realigning copper fibers under pressure can be seen if the specimens used in this study are closely examined. It is also noted that the realignment is more predominant in the 2/a ratios of 4, 8, and 25. In the epoxy matrix, the copper sample with the  $\mathcal{L}/a$  of 12.4, when compared with the corresponding

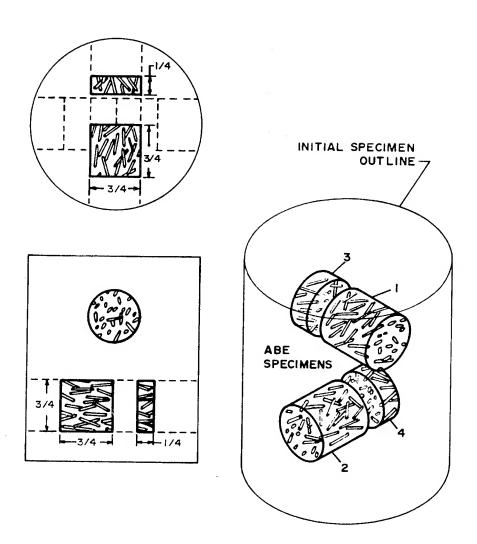


Figure 30. Anisotropic Specimens.

TABLE VIII

EXPERIMENTAL RESULTS OF THE ANISOTROPIC SPECIMENS

Sample	Density (gm cm <sup>-3</sup> )	Fiber Vol. Ratio (%)	K (W m <sup>-1</sup> deg <sup>-1</sup> )	T (°C)
ABE 1	4.16	41	10.5	60.9
ABE 3	4.20	42	15.2	59.1
ABE 2	4.16	39.1	10.23	40.5
ABE 4	4.27	40.5	9.74	40.5

aluminum one, has a thermal conductivity 16% higher with the epoxy matrix and 115% higher with the epoxy-Ti matrix; it could be possible, then, to conclude that the  $\mathcal{L}/a$  of 12.4 gives a better or favorable fiber distribution than the other ratios.

The aluminum fibers, when pressed, showed some alignment of the fibers perpendicular to the applied pressure for the  $\mathcal{L}/a$  of 4 in the epoxy matrix and for the  $\mathcal{L}/a$  of 8 in the epoxy-Ti matrix. (This alignment is not as pronounced as the one for the copper fibers.) For the other ratios, the aluminum fibers would bend and not relocate themselves as the copper ones. Note that the best fiber geometry for the aluminum fibers is the one with an  $\mathcal{L}/a$  ratio of 25 as shown in Figure 27.

The discussion of the experimental results has been based on just two similar samples (the 1/4" and the 3/4"). In order to draw acceptable conclusions, another experiment was performed. Six 3/4 of an inch long and ten 1/4 of an inch long samples were chosen at random from a group of twenty, and a statistical analysis was carried out to test the repeatability or replication of results. The experimental data for this experiment are shown in Tables IX and X.

With the aid of Appendix B, it is found that the mean and standard deviation of the conductivities, for the 3/4 of an inch samples, were 6.265 W m<sup>-1</sup> deg<sup>-1</sup> and 0.254, and for the 1/4 of an inch sample,  $\overline{X}$  = 4.27 W m<sup>-1</sup>deg<sup>-1</sup> and  $\overline{V}$  = 0.534. This clearly indicates that from the degree of consistency of the readings and reproducibility of the results, they are felt to be truly representative of the particular samples tested.

Table XI shows thermal conductivity measurements on four aluminum specimens having 100% fibers and four fiber geometries, and four copper specimens having 100% fibers and four fiber geometries.

TABLE IX  $\mbox{EXPERIMENTAL RESULTS OF "A$_1$, C$_1$, D$_1$, E" SAMPLES (1/4" THICK, MEASURED ) }$ 

		AT 40.5°C)	
Specimen No.	Density (gm cm <sup>-3</sup> )	Fiber Vol. Ratio (%)	$(W m^{-1} deg^{-1})$
1	2.00	39.0	3.84
2	1.98	37.5	4.0
3	1.98	37.5	4.67
4	2.01	39.0	4.16
5	1.96	35.9	4.79
6	1.98	37.5	4.22
7	1.99	38.2	4.51
8	2.03	41.4	5.12
9	1.99	38.2	3.24
10	2.03	41.4	4.16

TABLE X  $\mbox{EXPERIMENTAL RESULTS OF "A$_1$, C$_1$, D$_1$, E" SAMPLES (3/4" THICK) }$ 

Specimen	Density (gm cm <sup>-3</sup> )	Fiber Vol. Ratio (%)	(W m <sup>-1</sup> deg <sup>-1</sup> )	T (°C)
1	2.08	35.6	7.02	60.8
2	2.07	35.4	5.69	61.0
3	2.03	34.0	5.87	60.6
4	2.05	34.8	5.81	62.1
5	2.04	34.4	6.78	59.8
6	2.06	35.3	6.4	60.3

EXPERIMENTAL RESULTS OF SPECIMENS HAVING 100% I IBER VOLUME RATIO

TABLE XI

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1) $0.005$ "x $0.005$	Cross-sectional	Thermal Cond. (W m-1 deg-1) 10.0 8.2 17.3 15.6 15.6 10.9	Density (gm/cm3) 2.65 2.66 2.63 2.65 2.65 2.65 8.50	9.88 9.78 9.78 9.55 9.91	COPPER SAMPLES  2.867  2.855  2.855  2.855  2.855	(cm) 1.298 1.287 1.369 1.327 1.227	1) 0,005"x0,005"x0,062" 2) 0,005"x0,005"x0,125" 3) 0,015"x0,015"x0,062" 4) 0,015"x0,015"x0,125" 1) 0,005"x0,005"x0,062"
	Thickness Area Weight Density (cm) (cm) (cm) (cm) (gm) (gm/cm <sup>3</sup> ) (cm) (gm) (gm/cm <sup>3</sup> ) (cm) 1.298 2.867 9.88 2.65 1.287 2.860 9.55 2.63 1.309 2.855 9.91 2.65  2.65	11.2	8.44	29.87	2,880	1.228	3) 0.015"x0.015"x0.062" 4) 0.015"x0.015"x0.125"
N V	Thickness Area Weight Density (cm) (gm) (gm/cm <sup>3</sup> ) 1.298 2.867 9.88 2.65 1.287 2.855 9.78 2.66 1.269 2.855 9.78 2.66 1.309 2.855 9.91 2.65	:∠!	o/i	2	COPPER SAMPLES	إىب	
COPPER SAMPLES	Thickness Area Weight Density (cm) (gm/cm <sup>3</sup> ) (gm) (gm/cm <sup>3</sup> ) 1.298 2.867 9.88 2.65 1.287 2.855 9.78 2.66 1.269 2.860 9.55 2.63	15,6	2,65	9,91	2,855	1,309	15"x0,015"x0,125"
1,309 2,855 9,91 COPPER SAMPLES	Thickness Area Weight Density (cm <sup>2</sup> ) (gm) (gm/cm <sup>3</sup> ) 1.298 2.867 9.88 2.65 1.287 2.855 9.78 2.66	17,3	2,63	9,55	2,860	1,269	15"x0,015"x0,062"
1,269 2,860 9,55 1,309 2,855 9,91 COPPER SAMPLES	Thickness Area Weight Density (cm) (cm <sup>2</sup> ) (gm) (gm/cm <sup>3</sup> ) (1.298 2.867 9.88 2.65	8,2	2,66	9.78	2,855	1.287	05"x0,005"x0,125"
1.287 2.855 9.78 1.269 2.860 9.55 1.309 2.855 9.91 COPPER SAMPLES	Thickness Area Weight Density (cm) $(cm^2)$ $(gm)$	10.0	2,65	9.88	2,867	1,298	05"x0,005"x0,062"
1.298       2.867       9.88         1.287       2.855       9.78         1.269       2.860       9.55         1.309       2.855       9.91         COPPER SAMPLES		Thermal C (W m-1 de	Density (gm/cm <sup>3</sup> )	(gm)	Area $(cm^2)$	(Cm)	

These specimens were made per equation (82), i.e.,  $W_f = f V_T V_R$ , where  $V_{\mathrm{p}}$  is equal to 1. After the fibers were poured into the mold, a pressure of 29.4 tons was required to obtain the predetermined final dimensions. The densities of the specimens were found to be very close to the one of the pure metal (i.e., 2.78 gm  $cm^{-3}$  for aluminum and 8.92 gm  $cm^{-3}$  for copper); this indicates that the 100% fiber volume ratio was obtained. At this point, a very important observation is made in regard to the thermal conductivity measurements: the highest experimental  $K_{\mbox{\scriptsize R}}$  for the aluminum and copper specimens are 17.3 and 13.8 W m<sup>-1</sup> deg<sup>-1</sup> respectively; the discrepancy is large when these values are compared with the conductivities of an isotropic and homogeneous aluminum specimen ( $K_B = 210$ W m<sup>-1</sup> deg<sup>-1</sup>), or copper specimen ( $K_B = 368 \text{ W m}^{-1} \text{ deg}^{-1}$ ) as obtained from standard materials handbooks or by the earlier theories depicted in Figures 31 through 34. This experiment clearly shows the differences in conductivities between the pure metals and specimens made per equation (82), i.e.,  $W_f = f_T V_T V_R$ . In the study of such specimens, other factors should be considered, for example: contact resistance between fibers, oxide films, and other physical parameters. The purpose of this experiment was to clarify the 100% fiber volume ratios of the earlier theories and possibly open other areas of research.

## C. Theoretical Results

Theoretical curves obtained by other authors are shown in Figures 31 through 34. These curves represent the composite thermal conductivity versus the fiber volume ratio. Equations 9, 10, and 11, were not plotted since they are empirical equations and do not check with the results obtained in this study. Equations 16 and 17 do not give meaningful results; for instance, with the  $K_{\mathrm{f}}$  and  $K_{\mathrm{m}}$  used in this study, a zero-division is experienced whenever volume ratios from zero to 100% are substituted. In obtaining the curves in Figures 31 thru 34, it seems that it was mathematically convenient for the authors to assume high symmetry in representing a composite structure; for instance. filamentary composites were represented by square lattices, rectangular lattices, lamellar lattices, tetragonal lattices, cubic lattices, and close packed structures were represented by hexagonal lattices, face centered cubic etc. Equations obtained by such assumptions have rigorous values of composite thermal conductivity depending, in general, on the fiber volume ratio  $(v_{R_{\bar{1}}})$ , the thermal conductivities of the phases  $(K_m, K_f)$ , and/or some parameter "F" determined by the mode of packing, or on some empirical parameter "n". Figures 26 through 29 definitely show that the factors involved in describing a composite structure are not only  $V_{R_1}$ ,  $K_f$  and  $K_m$ , but also the fiber geometry and its distribution.

Curves 1 (Equations 2 and 22) and 7 (Equation 3) are plotted to show the upper and lower bounds for the thermal conductivity of composites as obtained by earlier theories. The stochastic thermal model developed in this study includes the fiber geometry and its distribution. The discussion of this model can be found in Part II, Part IV, and Appendix A.

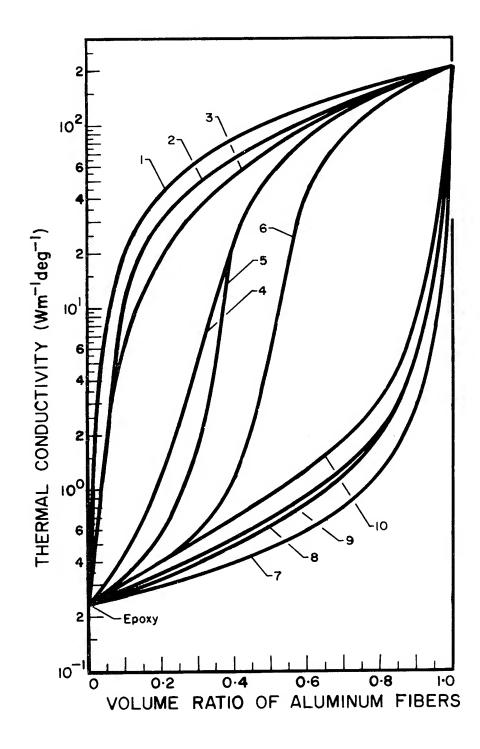


Figure 31. Thermal Conductivity vs. Fiber Volume Ratio (Al-Ep). 1. Maximum Conductivity (Equations 2 and 22). 2. (Equation 12). 3. (Equation 6). 4. Random Mixture (Equation 14). 5. (Equations 5 and 21). 6. (Equation 7). 7. Minimum Conductivity (Equation 3). 8. (Equation 16). 9. (Equation 18). 10. (Equation 19).

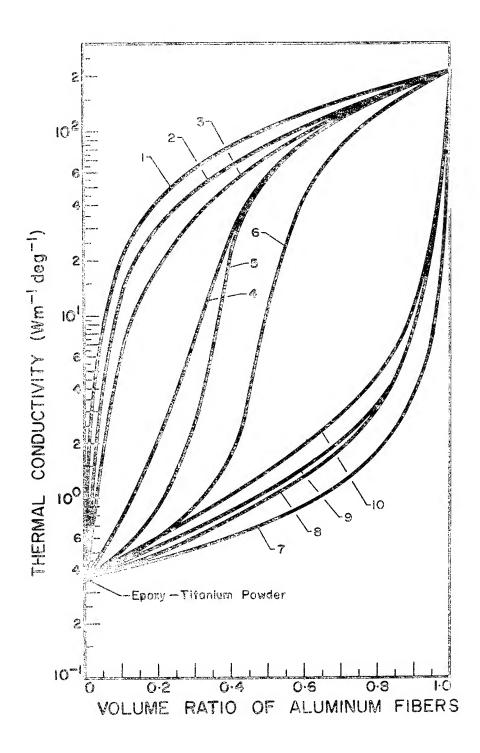


Figure 32. Thermal Conductivity vs. Fiber Volume Ratio (Al-Ep-Ti). 1. Maximum Conductivity (Equations 2 and 22). 2. (Equation 12). 3. (Equation 6). 4. Random Mixture (Equation 14). 5. (Equations 5 and 21). 6. (Equation 7). 7. Minimum Conductivity (Equation 3). 8. (Equation 16). 9. (Equation 18). 10. (Equation 19).

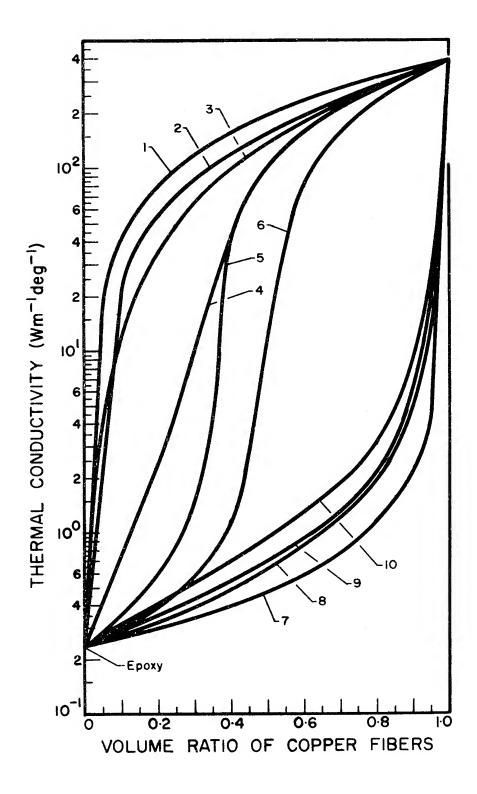


Figure 33. Thermal Conductivity vs. Fiber Volume Ratio (Cu-Ep). 1. Maximum Conductivity (Equations 2 and 22). 2. (Equation 12). 3. (Equation 6). 4. Random Mixture (Equation 14). 5. (Equations 5 and 21). 6. (Equation 7). 7. Minimum Conductivity (Equation 3). 8. (Equation 16). 9. (Equation 18). 10. (Equation 19).

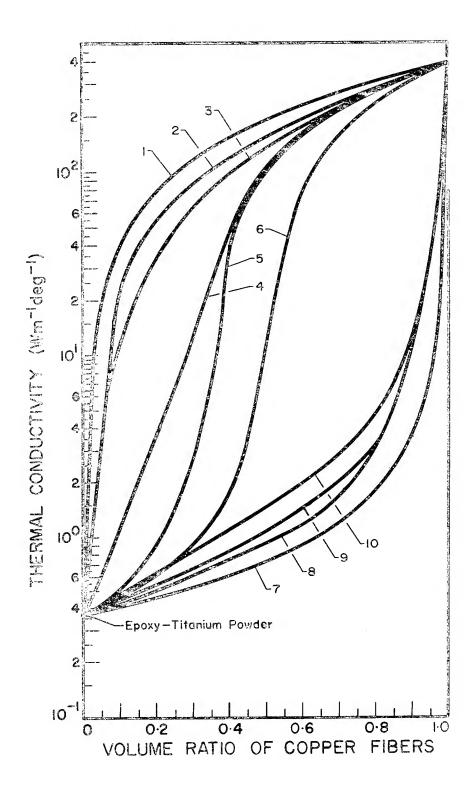


Figure 34. Thermal Conductivity vs. Fiber Volume Ratio (Cu-Ep-Ti). 1. Maximum Conductivity (Equations 2 and 22). 2. (Equation 12). 3. (Equation 6). 4. Random Mixture (Equation 14). 5. (Equations 5 and 21). 6. (Equation 7). 7. Minimum Conductivity (Equation 3). 8. (Equation 16). 9. (Equation 18). 10. (Equation 19).

Theoretical results obtained from this model are limited by the size of the computer, as it will be explained in section D of Part IV.

The curves for the  $\frac{l}{a}$  ratios of 4, 8, 12.4 and 25 plotted in Figures 26 through 29, have been obtained from the stochastic model. Note the curve  $\frac{l}{a}$  = 8: this curve is made of two parts, the section from 0 to 12.8% fiber volume ratio was obtained from the computer program in Appendix A and Figure 8, while from 20 to 55% was obtained from the theory of Appendix D and the thermal model of Figure 8. The reason for choosing the  $\frac{l}{a}$  = 8 is the following one: from the fiber volume ratio equation, i.e.,  $V_R = N V_f/V_T$ , it is noted that the number of fibers (N) needed to fill a specific volume ( $V_T$ ) is  $N = (V_R/V_f) V_T$ , hence for a constant  $V_T$ , N will increase as  $V_f$  will decrease. The  $V_R/V_f$  used in this study are listed in the second column of Tables XIV through XVII. Note also that the least amount of fibers needed to fill a predetermined volume  $V_T$  with a prescribed fiber volume ratio ( $V_R$ ) is the fiber with the  $\frac{l}{2}$  = 8, followed by the  $\frac{l}{2}$  = 4, 25, and 12.4 respectively.

The Mark II, G.E. computer memory bank, allowed only 500 fibers, or a  $V_R$  of 12.8% (see section D of Part IV) no matter what the fiber geometry, hence if the  $\frac{1}{a}$  of 4 were chosen, 500 fibers would have given a fiber volume ratio of 6.4%; the  $\frac{1}{a}$  of 25, a  $V_R$  of 1.42%; and the  $\frac{1}{a}$  of 12.4, a  $V_R$  of 0.71%. The thermal conductivities for the aforementioned fiber volume ratios and geometries could have been calculated by the computer, but the answers would have been very close or probably the same as the matrix: for this reason the  $\frac{1}{a}$  of 8 was chosen.

Work is continuing in refining the computer program and rewriting the program for a larger computer such as the IBM 360. Because of the

computer limitations, another theory was investigated. See Appendix D. The results from this theory are plotted in Figures 26 through 29. The thermal conductivities obtained from this theory follow the experimental behavior of the specimens, i.e.,  $K_{\rm B}$  is a function of the fiber volume ratio and fiber geometry. The question arises: Is this theory good for all fiber volume ratios? By looking at the trend of the experimental data we can certainly make some qualitative conclusions; for example, Figures 26 through 29 show that for the  $\frac{2}{a}$  of 8 this theory is good between 20% to 40% or perhaps higher; for the  $\frac{2}{a}$  ratios of 4, 12.4, and 25 the theory could be useful for fiber volume ratios higher than 40%. The computer program of Appendix A is believed to be correct; the limited data obtained from it seems to be directed toward the experimental values, only a larger computer could ascertain its validity.

## D. Computer Program

The computer program is written for the Mark II G.E. time sharing system. The program is sufficiently versatile so that it can be run by anyone. To gain access to the computer, one depresses the ORIG button on the teletypewriter console and dials the telephone number of the central computer. The computer then responds with a series of statements. The underlined portions are user supplied while the remainder are computer responses.

USER NUMBER - - - SBT43111, THMRDE

PROJECT ID --- FIBERS

SYSTEM - - - - FOR

NEW OR OLD - - - - OLD

OLD FILE NAME - - - COMP 1

READY

RUN

COMP 1

09.38

03/28/69

FORT-C-F DATED 10 MAR 1969

SAMPLE SIZE INCREMENT? 10

(The number 10 is typed here to generate 10 fibers into the specified model volume).

FOR SAMPLE SIZE = 10 DISCARDED FIBERS 0

(This statement tells the user that no fibers intersected any other fiber when introduced in the matrix. NOTE: The fibers intersecting the model's surface are rejected, but are not counted in "DISCARDED FIBERS".)

MORE FIBERS? 0

(The user types 0 or 1 if his answer is "yes" or "no" respectively.)

SAMPLE SIZE INCREMENT? 20

FOR SAMPLE SIZE = 20 DISCARDED FIBERS 2

(Here 2 new fibers intersected with one of the previous fibers; therefore they were discarded and two new ones were generated in their place.) MORE FIBERS? 1

FIBER DENSITY CHECK? 0

(Again, the user types 0 or 1 for yes or no. If the answer is yes, the user will now perform the heat conduction problem. At first the model is subdivided in a number of heat channels, hence:

VALUES FOR DY AND DX? .015, .015

(Here the user states the size of the integrating unit or heat channel area that is desired to be used. In this case, an area of .000225 is used to estimate the fiber density, since the fiber used in the analysis has a cross section of 0.015 X 0.015 square inches.)

FIBER HITS = 1.0300E + 02

MATRIX HITS = 1.8730E + 03

SUMKB = 2.9037E - 01

SUMKM = 4.4578E + 00

AVERAGE KB = 2.4029E - 03

(FIBER HITS represent the number of heat channels where fibrous material was located. MATRIX HITS represent the number of heat channels where fibrous material was not located. SUMKB represents the sum of the thermal conductivities of the FIBER HITS. SUMKM represents the sum of the thermal conductivities of the MATRIX HITS. Note that (MATRIX HITS) times ( $K_m$ ) should be equal to SUMKM, in this case  $K_m = 0.00238 \; W \; cm^{-1} \; deg^{-1}$ . AVERAGE  $K_B$  is the total thermal conductivity of the matrix with the specified

number of fibers or the specified fiber volume ratio.)

FIBER DENSITY CHECK? 1

(If the answer were 0, the computer would again respond with a request for: "VALUES FOR DY AND DX?", thus enabling the user to change the integrating interval if desired.)

MORE FIBERS? STOP

(The program is terminated with this response. If more fibers were desired and the proper response typed, the user could go through the aforementioned cycle again. NOTE: The program could be terminated at any response by typing STOP or typing ANY NUMBER GREATER THAN ONE AT "MORE FIBERS?").

This computer program was unable to generate more than 500 fibers since the program required a considerable amount of computer memory, and the Mark II G.E. Time sharing system was unable to furnish the proper amount.

Tables XII and XIII present computer results for the average thermal conductivity of a model having a geometry similar to the "ABC" specimen used in the "COLORA" instruments. These results were plotted in Figure 26 for comparison with the experimental ones. Some of the disadvantages of this program are the limitation on the fiber volume ratio and the cost of the computer time; for instance, the running time for Table XIII was 1664 units, the terminal time was 146 units. One unit costs 30 cents; hence 1810 units cost \$543.00! (Note that one unit is equivalent to 4/3 of a second), or Table XII cost \$2172.00.

TABLE XII

COMPUTER RESULTS FOR FIBER: 0.015" x 0.015" x 0.125"

 $K_B (W m^{-1} deg^{-1})$ 

			_		
No. of Fibers	V <sub>R</sub> (%)	A1-Ep	Al-Ep-Ti	Cu-Ep	Cu-Ep-Ti
0	0	0.238	0.365	0.238	0.365
20	0.51	0.240	0.367	0.240	0.367
100	2.56	0.252	0.370	0.252	0.370
200	5.12	0.376	0.441	0.456	0.520
300	7.68	0.503	0.549	0.663	0.709
350	8.98	0.517	0.557	0.677	0.714
500	12.80	0.923	0.945	1.334	1.356

### TABLE XIII

### EXAMPLE OF COMPUTER PROGRAM OUTPUT

COMP1 15:17 04/11/69 FORT-C-F DATED 10 MAR 69

SAMPLE SIZE INCREMENT?100

FOR SAMPLE SIZE = 100 DISCARDED FIBERS 14
MORE FIBERS?1

FIBER DENSITY CHECK?0

FIBER CONDUCTIVITY CONSTANT?2.1

VALUES FOR DY AND DX?.015,.015

FIBER HITS = 5.7700E+02 MATRIX HITS = 1.3990E+03 SUMKB = 1.6649E+00 SUMKM = 3.3296E+00 AVERAGE KB = 2.5276E-03 FIBER DENSITY CHECK?1

MORE FIBERS?0

SAMPLE SIZE INCREMENT?400

FOR SAMPLE SIZE = 500 DISCARDED FIBERS 843
MORE FIBERS?1

FIBER DENSITY CHECK?O

FIBER CONDUCTIVITY CONSTANT?2.1

VALUES FOR DY AND DX?.015,.015 FIBER HITS = 1.6250E+03 MATRIX HITS = 3.5100E+02 SUMKB = 1.7414E+01 SUMKM = 8.3538E-01 AVERAGE KB = 9.2354E-03 FIBER DENSITY CHECK?0

FIBER CONDUCTIVITY CONSTANT?3.68

VALUES FOR DY AND DX?.015,.015 FIBER HITS = 1.6250E+03 MATRIX HITS = 3.5100E+02 SUMKB = 2.5522E+01 SUMKM = 8.3538E-01 AVERAGE KB = 1.3339E-02 FIBER DENSITY CHECK?1

MORE FIBERS?10

PROGRAM STOP AT 1520 USED 1373.28 UNITS

# E. Distribution of Fibers in a 3-Dimensional Space

The Mark II G.E. computer was unable to furnish the proper computer memory bank for fiber volume ratio higher than 12.8. In Appendix D, an analysis is presented to determine the mean distance, between an individual fiber and its nearest neighbor. This distance is equivalent to a mean amount of matrix material between a fiber and its nearest neighbor in a collection of fibers randomly distributed in a 3dimensional space. It is noted that in the expression for  $\delta$  , the fiber volume ratio and the fiber geometry play a very important role in the later determination of the composite thermal conductivity. Results obtained from this approach, for the 20 and 40 percent fiber volume ratio, seem to have the same behavior as the experimental results and, in some cases, for the aluminum fibers with an L/a of 8, the match is very good. Tables XIV through XVII show theoretical results obtained from this theory for volume ratios of 20, 30, 40, 50 and 60% for the different geometries and materials used in this study. NOTE: This model does not work for volume ratios of 0 and 100% (see the expression for S in Appendix D). Probably more reliable theoretical models could be developed to predict the thermal conductivity of the type of composites used in this study. The experimental work indicates the general behavior of the thermal conductivity of composites as a function of  $K_f$ ,  $K_m$ ,  $V_R$ , the fiber geometry, and its distribution; this data should be very helpful in developing better and more reliable models. Because of the many variables encountered, it seems almost impossible to approach this problem from a deterministic point of view. In this study an attempt was made to develop a statistical or stochastic model, the theoretical and experimental results seem to indicate the feasibility of the statistical and probabilistic approach.

TABLE XIV

THERMAL CONDUCTIVITY OF FIBERS RANDOMLY DISTRIBUTED IN A 3-D SPACE FIBER: 0.005" X 0.005" X 0.062"

Fiber Vol.	$\frac{v_R}{v_f}$	8		*K <sub>B</sub> (W m	1 deg-1)	
(%)	(cm-3)	L	A1-Ep	Al-Ep-Ti	Cu-Ep	Cu-Ep-Ti
20	7874	0.043	5.29	8.02	5.35	8.15
30	11811	0.038	6.04	9.13	6.11	9.30
40	15748	0.034	6.63	10.00	6.72	10.21
50	19685	0.032	7.12	10.74	7.23	10.97
60	23622	0.030	7.55	11.37	7.67	11.64

 $<sup>*</sup>K_{B}$  = Thermal Conductivity

TABLE XV

THERMAL CONDUCTIVITY OF FIBERS RANDOMLY DISTRIBUTED IN A 3-D SPACE FIBER: 0.005" X 0.005" X 0.125"

Fiber Vol. Ratio	$\frac{v_R}{v_f}$	8		*K <sub>B</sub> (W m	1 deg-1)	
(%)	(cm-3)	L	A1-Ep	Al-Ep-Ti	Cu-Ep	Cu-Ep-Ti
20	3906	0.055	4.21	6.40	4.25	6.48
30	5859	0.048	4.81	7.29	4.86	7.40
40	7812	0.044	5.28	8.00	5.34	8.13
50	9765	0.041	5.68	8.59	5.74	8.74
60	11719	0.038	6.03	9.11	6.10	9.27

 $<sup>*</sup>K_B$  = Thermal Conductivity

TABLE XVI

THERMAL CONDUCTIVITY OF FIBERS RANDOMLY DISTRIBUTED IN A 3-D SPACE FIBER: 0.015" X 0.062"

Fiber Vol. Ratio	VR V£	8_		*K <sub>B</sub> (W m	-1 deg-1)	
(%)	$(cm^{\frac{1}{2}3})$	I.	Al-Ep	Al-Ep-Ti	Cu-Ep	Cu-Ep-Ti
20	874.5	0.092	2.58	3,93	2.59	3.96
30	1311.8	0.079	2.95	4.49	2.96	4.53
40	1749.0	0.072	3.24	4.95	3.26	4.98
50	2186.3	0.067	5.49	5.30	3.51	5.36
60	2623.5	0.063	3.70	5.63	3.73	5.69

 $<sup>*</sup>K_B$  = Thermal Conductivity

TABLE XVII

THERMAL CONDUCTIVITY OF FIBERS RANDOMLY DISTRIBUTED IN A 3-D SPACE FIBER: 0.015" X 0.125"

Fiber Vol.	$v_R$	8		*K <sub>B</sub> (W m	1 deg-1)	
(%)	(cm <sup>-3</sup> )	L	A1-Ep	Al-Ep-Ti	Cu-Ep	Cu-Ep-Ti
20 30 40 50	434.8 652.2 869.6 1087.0 1304.3	0.115 0.100 0.091 0.085 0.080	2.05 2.34 2.57 2.77 2.94	3.13 3.57 3.93 4.22 4.48	2.06 2.35 2.59 2.78 2.96	3.15 3.60 3.95 4.26 4.52

 $<sup>*</sup>K_B$  = Thermal Conductivity

#### PART V

#### SUMMARY OF RESULTS AND CONCLUSIONS

- 1. The special technique used in the fabrication of the models is very effective for fiber volume ratios of 20 to 50 percent.

  Because of the vacuum technique, very few pits or holes are present in the specimens having 20% ratios or lower, and practically none for higher ratios. Individual materials can be prepared with reproducible properties.
- 2. The testing procedure used in this study, i.e., the "Colora" and the "Comparative Apparatus", is suitable for evaluation of the thermal conductivity of the type of composites used in this study and would seem to indicate that accuracies of better than  $\pm$  3% for the "Colora" and  $\pm$  2% for the "Comparative Apparatus" can be obtained.
- 3. Effective thermal conductivities were measured for two factorial experiments comprising thirty two specimens each. With the aluminum fibers embedded in an epoxy matrix, the conductivities ranged from 0.95 to 4.61 W m<sup>-1</sup> deg<sup>-1</sup>. With the copper fibers and epoxy matrix, the conductivities ranged from 1.04 to 2.66 W m<sup>-1</sup> deg<sup>-1</sup>. With the aluminum fibers and the epoxy-titanium powder matrix, the conductivities ranged from 1.23 to 12.05 W m<sup>-1</sup> deg<sup>-1</sup>. With the copper fibers and the epoxy-titanium powder matrix, the conductivities ranged from 1.79 to 10.05 W m<sup>-1</sup> deg<sup>-1</sup>.
- 4. Two statistical analyses; one for the 1/4" specimens, comprising ten samples, and the other, for the 3/4" sample, comprising six samples, were performed. The fiber volume ratios for the 1/4" samples' (having the

epoxy-titanium powder matrix and aluminum fibers) ranged from 37.5 to 41.4 percent, and their measured thermal conductivities ranged from 3.24 to 4.79 W m<sup>-1</sup> deg<sup>-1</sup>. The fiber volume ratio for the 3/4" samples' (having epoxy-titanium powder matrix and aluminum fibers) ranged from 34 to 35.6 percent, and their measured thermal conductivities ranged from 5.81 to 7.02 W m<sup>-1</sup> deg<sup>-1</sup>. For the 3/4" specimens, the mean thermal conductivity is 6.25 W m<sup>-1</sup> deg<sup>-1</sup>, and the standard deviation  $\mathcal T$  is 0.595. For the 1/4" specimens, the mean thermal conductivity is 4.27 W m<sup>-1</sup> deg<sup>-1</sup>, and the standard deviation  $\mathcal T$  is 0.594.

- 5. Effective thermal conductivities were measured for four specimens (two 1/4" and two 3/4") having anisotropic characteristics.

  The specimens were made of copper fibers embedded in an epoxy-matrix. The fiber volume ratios of the four specimens ranged from 39.1 to 42 percent, and their measured thermal conductivities ranged from 9.74 to 15.2 W m<sup>-1</sup> deg<sup>-1</sup>.
- 6. Effective thermal conductivities were measured for four aluminum specimens having the same fiber volume ratios (100%) and four fiber geometries; and four copper specimens having the same fiber volume ratios (100%), and four fiber geometries. Their densities ranged from 2.63 to 2.66 gm cm<sup>-3</sup> for the aluminum, and from 8.44 to 8.50 gm cm<sup>-3</sup> for copper. Their measured conductivities ranged from 8.2 to 17.5 W m<sup>-1</sup> deg<sup>-1</sup> for aluminum specimens and from 10.9 to 13.8 W m<sup>-1</sup> deg<sup>-1</sup> for the copper specimens.
- 7. The most meaningful factor to be considered for an optimum design of the type of composites used in this study are, in the order of importance, the conductivity of the matrix, the fiber volume ratio, and the fiber cross section as obtained from the factorial experiment.

- 8. Anisotropic characteristics for a copper fiber composite can be achieved for fiber volume ratios of 40% or higher by just pressing the fibers into a predetermined volume.
- 9. The thermal conductivity of composite materials, made of highly conductive fibers randomly distributed in low conductive matrices, is not only a function of the fiber volume ratio, the conductivity of the fiber, the conductivity of the matrix as covered by earlier theories, but also a function of the fiber geometry and its distribution. Earlier theoretical efforts consisted of mathematical models which are highly idealized (Maxwell, Rayleigh, Bruggman, etc.); most of these models either underestimate or overestimate the thermal conductivities of the type of composites presented in this study.
- matrix filled with fibers, ellipsoidal or cylindrical in shape and randomly distributed according to a prescribed fiber volume ratio, have been derived. With these equations, a stochastic model has been proposed which allows one to simulate realistically composite structures. Limited theoretical predictions of the stochastic model seem to check with the experimental data, and it shows that additional factors are involved in describing composites which are not covered by earlier theories, i.e., fiber geometry and its distribution. Theoretical results of composite thermal conductivity, up to a fiber volume ratio of 13 percent, are obtained with the aid of a high speed computer. Higher fiber volume ratios can be obtained if a computer with larger memory bank is supplied.
- 11. Theoretical predictions of the composite thermal conductivity of fiber volume ratios of 20 to 60 percent are obtained by distributing

fibers in a 3-dimensional space in accordance with a Poisson process. This theory was investigated because of the computer limitations of preceding paragraph 10. Experimental results for the fiber geometry of  $\frac{L}{a} = 8$  seem to check very well with this theory; for  $\frac{L}{a}$  of 4, 12.4 and 25, this theory could be used for fiber volume ratios higher than 40%.

- 12. It was found that a specific specimen formulation (sample ACED) gives a thermal conductivity similar to that of stainless steels but a density only a quarter of that of the steels.
- 13. The experimental work indicates the general behavior of the thermal conductivity as a function of  $K_f$ ,  $K_m$ ,  $V_R$ , fiber geometry, and its distribution. The data obtained is reliable, and it is recommended to use this data for guidance in developing better, simple, and more reliable models.

#### PART VI

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#### APPENDIX A

#### COMPUTER PROGRAM

The computer program presented in this appendix has been designed for fibers with cylindrical cross sections and comprises the following tasks:

- 1. Generates fibers in space per theory outlined in Part II.
- 2. When the desired number of fibers is generated, a "FIBER DENSITY CHECK" is made. This is done by dropping a Z-line perpendicular to the base of the model (Figure 35). The amount of fibrous material intersected by this line is calculated as follows:
- a. The distance of a fiber to the  $\Xi_1$ -line is first determined. This is done by projecting the fiber on to the x-y plane and taking the  $\Xi_1$ -line as a point  $P_L$ . Since the coordinates of  $P_L$  and the equation of the fiber in 2-space are known, the perpendicular distance d can be easily determined by:

$$d = \frac{|Ax_1 + By_1 + C|}{\sqrt{A^2 + B^2}}$$
 (A-1)

where  $(x_1, y_1)$  are the coordinates of  $P_L$  and Ax + By + C is the equation of the fiber projection. If d is greater than the radius of the fiber, there is no intersection between the  $\mathbb{F}_1$ -line and the fiber. If d is less than the radius, the problem must be examined more carefully.

b. The coordinates of the intersection must now be found and checked to see if they lie within a range slightly larger than the

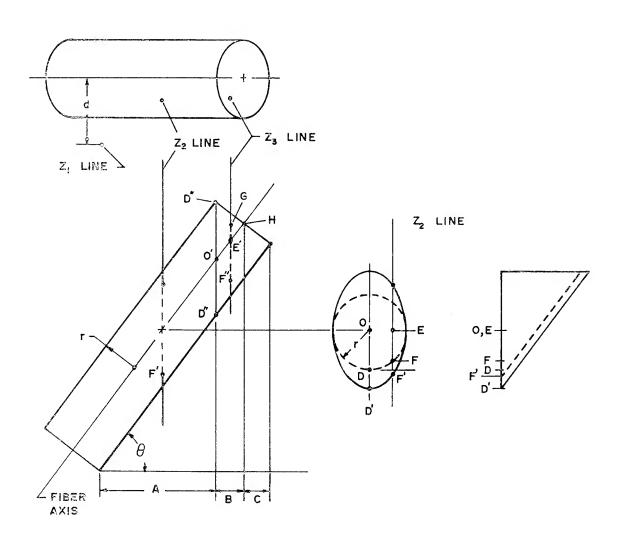


Figure 35. Diagram of a Cylindrical Fiber.

fiber projection. If the coordinates lie outside the range, no problem exists. If within the range, the following possibilities are to be scrutinized.

(1) If the  $\mathbb{Z}_2$ -line is contained within "A" the calculation of the fibrous material from Figure 35 is:

$$EF' = \frac{E F}{\cos \theta} \tag{A-2}$$

where EF = 
$$\sqrt{\overline{OF}^2 - \overline{OE}^2}$$

and

OF = Fiber radius.

OE = Distance of the  $Z_2$ -line to the fiber axis.

Note that the other relations used to obtain EF' are:

$$\frac{OD'}{OD} = \frac{EF'}{EF} \tag{A-3}$$

where OD = OF

and 
$$OD^{\bullet} = \frac{r}{\cos \theta}$$
 (A-4)

(2) If the  $\Xi_3$ -line resides in B or C, a slight variation to A may be necessary as can be seen in Figure 35.

$$F''G = F''E' + E'G$$
 (A-5)

using similar triangles, OH D' and EHG:

$$\frac{E'G}{O'D''} = \frac{E'H}{O'H}$$

$$E'G = \frac{(E'H) \times (O'D'')}{O'H}$$
(A-6)

where 0'H = (fiber radius) X (tan 0)

E'H is a component of the undirected magnitude between the fiber extreme and also a component of the  $\Xi$ -line relative to the fiber.

(3) If the 
$$\Xi_3$$
-line is in C, then

$$F^{ii\dagger} G^{\dagger} = F^{ii\dagger} G^{\dagger} - G^{\dagger} E^{ii}$$
 (A-7)

The following computer listing is based on the aforementioned outline. A detailed discussion on how to use the program is presented in Part  ${\rm IV}$ , Section D.

```
100 COMMOND, DI, H, XL, START, COUNT
110 COMMONXM(500), XH(500), DELTX(500), C(500), Z(500), A(500), X(500)
120 COMMONR(500),S(500),T(500),U(500),SINP(500),COSP(500)
130 DIMENSIONB(500), Y(500)
135 COMMONDIAG, DIAG2, CNSTF, CNSTM
140CXL IS THE LENGTH OF THE FIBER
150CD IS THE DIAMETER OF THE FIBER
160CD1 IS THE DIAMETER OF THE CYLINDER
170CH IS THE HEIGHT OF THE CYLINDER
180C N IS THE SAMPLE SIZE
190C N1 REP THE NR OF SAMPLES
200 DATA ST, CT/12345.,0./
210 DATA N1/1/
220 DATA XLD, DDD, D1D/.125,.015,.750/
230 DATA HD/.250/
244 CNSTM= . 00238
250 N=0
260 START=ST
270 COUNT=CT
280 SUMB=0.
290 SUM1=0.
300 NADD=0
310 SUM2=0.
320 XL=XLD
330 D=DDD
340 D1=D1D
350 H=HD
360 DIAG=SQRT(XL*XL+D*D)
370 DIAG2=DIAG/2.
380 NNP2=2
390 NNP1=1
400 CALL FIBER(A(1),B(1),C(1),X(1),Y(1),Z(1),SINP(1),COSP(1),DELTX(1))
410 CALLEQUAT(A(1),B(1),C(1),X(1),Y(1),Z(1),R(1),S(1),T(1),U(1),
420&XM(1),XH(1))
430 1000PRINT, "SAMPLE SIZE INCREMENT"
440 INPUT, NN
450 N=N+NN
460 NT=0
470 D020I=NNP2,N
480 2CALLFIBER(A(I),B(I),C(I),X(I),Y(I),Z(I),SINP(I),COSP(I),
490&DELTX(I))
500 CALLEQUAT(A(I),B(I),C(I),X(I),Y(I),Z(I),R(I),S(I),T(I),U(I),
510&XM(I),XH(I))
520 J=I-1
530C P2, P3, P4 ARE COEFF OF P1P2
540 4 P2=A(I)-A(J)
550 P3=B(I)-B(J)
560 P4=C(I)-C(J)
570C Al, A2, A3 ARE COEFF OF V2
580 A1 = A(I) - X(I)
590 A2=B(I)-Y(I)
600 A3=C(I)-Z(I)
610C B1, B2, B3 ARE COEFF OF VI
620 B1=A(J)-X(J)
630 B2=B(J)-Y(J)
640 B3=C(J)-Z(J)
650C XN2, XN3, XN4 ARE COEFF OF V1XV2
```

```
COMP 1
          CONTINUED
                                                                         148
660 XN2=A2*B3-A3*B2
670 XN3=A3*B1-A1*B3
680 XN4=A1*B2-A2*B1
5900 D3 IS THE ABS VAL OF MAG VIXV2
700 D2=XN2*XN2*XN3*XN3+XN4*XN4
710 D3=SQRT(D2)
7200 PERP REP PERP. DIST BETWEEN CENTER LINE OF FIBERS
730 PERP=(P2*XN2+P3*XN3+P4*XN4)/D3
740 APERPSABS(PERP)
750 IF(APERP-D)17,31,31
760 17 XM2=B2*XN4-B3*XN3
770 XM3=B3*XN2-B1*XN4
780 XM4=B1*XN3-B2*XN2
7900 THE ABOVE REP THE COEFF OF NORMAL TO PLANE VIV3
600 C1=XM2*A(J)*XM3*B(J)*XM4*C(J)
810C XM2, XM3, XM4 C1 REP THE COEFF OF PLANE VIV3
820 C2=A2*A(I)-A1*B(I)
830 C3=A3=A(I)-A1*C(I)
8400 Cla C2a C3 REP CONSTANTS OF THE PLANE AND THE LINE INTERSECTING THE
850C PLANE.
            THE FOLLOWING IS THE SOLUTION OF THE 3 EQ.
860 D9=A3*A1*XM4+A1*A1*XM2+A1*A2*XM3
870 S1=(XM3*A1*C2+A1*A1*C1+A1*C3*XM4)/D9
880 S2*(XM4*(A2*C3-A3*C2)-A1*(XM2*C2-A2*C1))/D9
890 S3=(A3*(XM3*C2+A1*C1)-C3*(A1*XM2+A2*XM3))/D9
900 XN5=(XN2*PERP)/D3
910 XN6=(XN3*PERP)/D3
980 XN7=(XN4*PERP)/D3
930 SarS1+XN5
940 S5=S2+XN6
950 S6=S3+XN7
960 IF((A(J)-S1)*(X(J)-S1))19,19,31
970 19 IF((A(I)-S4)*(X(I)-S4))5,5,31
980 5 NT=NT+1
990 GGTG2
1000 31 IF(J-1)20,20,3
1010 3 J=J-1
1020 GOTO4
1030 20 CONTINUE
1040 PRINT, "FOR SAMPLE SIZE =",N,"
                                     DISCARDED FIBERS" NT
1050 900PRINT, "MORE FIBERS"
1060 INPUT, FIB
1070 IF(FIB-1.)1380,790,76
1080 790PRINT, "FIBER DENSITY CHECK"
1090 INPUT, DEN
1100 IF(DEN-1.)800,900,900
1102 800PRINT, "FIBER CONDUCTIVITY CONSTANT"
1103 INPUT, CNSTF
1110 PRINT, "VALUES FOR DY AND DX"
1120 INPUT, DY, DX
1132 NTOT=0
1134 NTOTL=0
1136 SUMB=0.
1138 SUM1=0.
1140 XCHAN2=DX/2.
1150 YCHAN2=DY/2.
```

1160 YZERO=0.

1170 YZERO=YZERO+YCHAN2-.375

1900 3 RETURN

1190 1190XZER0=XCHAN2 1200 XBQUN=SQRT( . 140625-YZERQ\*YZERQ) 1220 1175CALL CALCN(N,XZERO,YZERO,SUMB,SUM1,NTOT,NTOTL) 1355 1600XZERO=XZERO+DX 1360 IF(ABS(XZERO)-XBOUN)1175,1175,1601 1370 1601XZER0=XCHAN2 1380 XZERO=XZERO-DX 1390 1605CALL CALCN(N, XZERO, YZERO, SUMB, SUMI, NTOT, NTOTL) 1395 XZERO=XZERO-DX 1400 IF(ABS(XZERQ)-XBOUN)1605,1605,1603 1407 1603YZER0=YZER0+DY 1410 IF(YZERO-.375)1190,910,910 1420 910XNTQT=NTQT 1425 PRINT1426, XNTOT 1426 1426FORMAT(1X,12HFIBER HITS =,1P1E12.4) 1430 XTOTL=NTOTL 1435 PRINT1436, XTOTL 1436 1436FQRMAT(1X,13HMATRIX HITS =,1P1E12.4) 1437 PRINT1438, SUMB, SUM1 1438 1438FORMAT(1X,7HSUMKB =,1F1E12.4,2X,7HSUMKM =,1P1E12.4) 1440 SUMB=(SUMB+SUM1)/(XNTOT+XTOTL) 1450 PRINTIESS, SUMB 1460 1885FORMAT(1X,12HAVERAGE KB =,1P1E12.4) 1470 SUM2=SUM2+SUMB 1480 GOTO790 1490 1380NNP1=NNP1+NN 1500 NNP2=NNP1 1510 GDT01000 1520 76STOP 1530 END 1630 SUBROUTINE FIBER(A, B, C, X, Y, Z, SINP, COSP, DELTX) 1640 COMMON D.DI.H.XL.START, COUNT 1650 CALL RAND(START, COUNT, RND) 1660 2 R=SQRT(RND) 1670 R=D1/2.\*R 1680 CALL RAND(START, COUNT, RND) 1690 THETA=6.28318\*RND 1700 CALL RAND(START, COUNT, RND) 1710 C=H\*RND 1720 B=R\*SIN(THETA) 1730 A=R\*COS(THETA) 1750 CALL RAND(START, COUNT, RND) 1760 ALPHA=6.28318\*RND 1770 CALL RAND(START, COUNT, RND) 1780 SINP=RND 1785 COSP=SQRT(1.-SINP\*SINP) 1790 X=A+XL\*COS(ALPHA)\*COSP 1800 IF(ABS(X)-D1/2.)7,7,2 1810 7 Y=B+XL\*SIN(ALPHA)\*COSP 1820 IF(ABS(Y)-D1/2.)8,8,2 1840 8Z=C+XL\*SINP 1850 IF(Z-H)9,9,2 1860 9 IF(Z)2,11,11 1870 11DELTX=ABS((D/2.)\*SINP\*COS(ALPHA)) 1880 IF(DELTX-XL)3,3,4 1890 4DELTX=XL

1910 END 1920 SUBROUTINE EQUAT(A,B,C,X,Y,Z,R,S,T,U,XM,XH) 1930C THIS ROUTINE YIELDS THE EQ OF PROJ ONTO XY-PLANE IN THE FORM 1940C Y=MX+H 1950C THE EQUATION OF CENTER LINE OF FIBER IN THE FORM 1960C RX+SY+TZ+U=0. 1970 XM=(B-Y)/(A-X) 1980 XH=B-XM\*A 1990 A1=X-A 2000 B1=Y-B 2010 C1=Z-C 2020 R=B1+C1 2030 S=C1-A1 2040 T=-A1-B1 2050 C2=A1\*B-B1\*A 2060 C3=B1\*C-C1\*B 2070 C4=A1\*C-C1\*A 2080 U=C2+C3+C4 2090 RETURN 2100 END 2110 SUBROUTINE RAND(START, COUNT, RND) 2120 START=10000000./START 2130C CUT OFF FIRST THREE SIGNIFICANT DIGITS OF QUOTIENT 2140C TO GENERATE RANDOM NUMBER. 2150 IS=START 2160 FS=IS 2170 START=START-FS 2180 RND=START 2190 START=START\*100000. 2200 24 COUNT=COUNT+.1 2210 IF(COUNT-100000.) 4,3,3 2220 3 COUNT= · I 2230C ADD COUNT TO PREVENT REPETITIONS 2240 4 START=START+COUNT 2250C CHECK SIZE OF START 2260 IF(START-100000.)5,4,13 2270 5 IF(START-10000.)6,4,14 2280 6 IF(START-1000.)7,4,12 2290 7 IF(START-100.)8,4,11 2300 8 IF(START-10.)9,4,10 2310C MULTIPLY START BY APPROPRIATE CONSTANT TO OBTAIN NEW START 2320C WITH FIVE DIGITS BEFORE THE DECIMAL POINT. 2330 9 START=START\*10020.01 2340 RETURN 2350 10 START=START\*1000.2001 2360 RETURN 2370 11 START=START\*100.002 2380 RETURN 2390 12 START=START\*10.00002 2400 RETURN 2410 13 START=START/10. 2420 14 RETURN 2430 END 2440 SUBROUTINE CALCN(N, XZERO, YZERO, SUMB, SUM1, NTOT, NTOTL) 2445 COMMON D.DI.H.XL.START, COUNT 2450 COMMONXM(500),XH(500),DELTX(500),C(500),Z(500),A(500),X(500)

2460 COMMONR(500),S(500),T(500),U(500),SINP(500),COSP(500)

3050 SUMB=SUMB+CNSTB

2462 COMMONDIAG, DIAG2, CNSTF, CNSTM 2465 SUM=0. 2490 DQ40I=1.N 2500 DIST=ABS(XM(I)\*XZERO-YZERO+XH(I))/SQRT(XM(I)\*XM(I)+1.) 2510 IF(DIST-D/2.)41,41,40 2520 41 SLOPE=XM(I) 2530 YINT=XH(I) 2540 XHPRM=YZERO+XZERO/SLOPE 2550 XVAL2=(XHPRM-YINT)/(SLOPE+1./SLOPE) 2560 XDELT=DELTX(I) 2570 IF(C(I)-Z(I))43,43,42 2580 42 XVAL=X(I)+XDELT 2590 AVAL=A(I)-XDELT 2600 GOTO1182 2610 43 XVAL=X(I)-XDELT 2620 AVAL=A(I)+XDELT 2630 1182 IF((XVAL2-XVAL)\*(XVAL2-AVAL))61,61,40 2640 61 YVAL=XHPRM-XVAL2/SLOPE 2650 ZVAL = -(R(I) \* XVAL 2 + S(I) \* YVAL + U(I)) / T(I)2660 DELPX=ABS(Z(I)-ZVAL) 2670 DELPX=DELPX/SINP(I) 2680 IF(XDELT-DELPX)72,72,71 2690 72 DELPX=ABS(C(I)-ZVAL) 2700 DELPX=ABS(DELPX/SINP(I)) 2710 71 XLOC=(2./XDELT)\*ABS(XZERO-(AVAL+XVAL)/2.)-(XVAL-AVAL)/XDELT+1. 2720 MLOC=XLOC 2730 CA=SQRT(D\*D/4.-DIST\*DIST) 2740 CAPRM=ABS(CA/COSP(I)) 2750 VERT=DELPX\*CAPRM/XDELT 2760 IF (VERT-DIAG2)83,83,84 2770 84 VERT=DIA62 2780 83 IF(MLQC)44,45,46 2790 44 IF(CAPRM-DIAG2)49,49,48 2800 48 CAPRM=DIAG 2810 49 CAPRM=2.\*CAPRM 2820 GOT054 2830 45 IF(VERT-CAPRM)51,51,52 2840 51 CAPRM=2.\*VERT ≈50 (0T054 2860 52 CAPRM=CAPRM+VERT 2870 GOTO54 2880 46 IF(VERT-CAPRM) 56,57,55 2890 56 CAPRM=CAPRM-VERT 2900 G) TO 54 2910 57 CAPRM=VERT 2920 G)T054 2930 55 CAPRM=VERT-CAPRM 2940 54 SUM=SUM+CAPRM 2950 40 CONTINUE 2960 IF(SUM-H)1720,1720,1730 2980 1730SUM=H 2990 1720IF(SUM)1670,1670,1660 3000 1660SUMM=SUM/H 3010 CNSTB=CNSTF\*CNSTM/(SUMM\*CNSTM+CNSTF\*(1.-SUMM)) 3030 200FORMAT(4E12.3) 3040 NTOT=NTOT+1

COMP1 CONTINUED

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3060 RETURN

3070 1670SUM1=SUM1+2.38E-3

3080 NTGTL=NT0TL+1

3090 RETURN

4000 END

#### APPENDIX B

#### DEFINITIONS OF STATISTICAL AND CHEMICAL TERMS

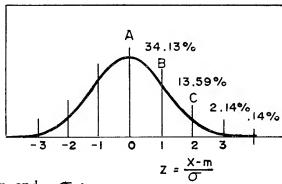
- Population: Any finite or infinite collection of individual things, objects, or events.
- 2. Sample: A portion of the population.
- 3. Distribution: Refers to the distribution of a characteristics or a group of characteristics in a population (it is usually obtained by measurements).
- 4. Random Sampling (Simple): This type of sampling is defined by the requirement that each individual in the population has an equal chance of being the first member of the sample; after the first member is selected, each of the remaining individuals in the population has an equal chance of being the second member of the sample; and so forth.
- 5. Selection of a Random Sample: It is advisable to use a table of random numbers as an aid in selecting the sample.
- 6. Properties of Distributions:

Normal or Gaussian distribution, symmetrical bell-shaped curve, is completely determined by:

- m, the  $\underline{\text{arithmetic}}$   $\underline{\text{mean}}$  (center of gravity) of the distribution and
  - $\sigma$  , the standard deviation (radius of gyration about m).
  - $\sigma^2$ , is the variance of the distribution (second moment about m).
- Z, is the distance from the population mean in units of  $\sigma$  and is  $\frac{(x-m)}{\sigma}$  where x represents any value in the population.

P, the proportion of elements in the population which have values of Z smaller than any given Z (P is found from table on page 549 of reference 28) if Z is known.

The following graph shows these percentages of the population in various intervals of Z. Example: 34.13% of the population will have values of Z between 0 and 1 (or 0 and -1).



7. Estimation of m and  $\sigma$ :

$$m = \overline{x} = \frac{1}{n} \sum_{i=1}^{n} x_i$$

$$\sigma_{x}^{2} = S^{2} = \frac{n \sum_{i=1}^{n} x_{i}^{2} - \left(\sum_{i=1}^{n} x_{i}\right)^{2}}{n (n-1)} =$$

$$(n-1) \sigma_{x}^{2} = S(x^{2}) - \frac{1}{n} \left[S(x)\right]^{2}$$

m and  $\sigma$  will differ from sample to sample.

8. Confidence Interval: An interval within which we estimate a given population parameter to lie (e.g., the population mean m with respect to some characteristic). Inasmuch as estimates of m and  $\sigma$  vary from sample to sample, interval estimates of m and  $\sigma$  may sometime be

preferred to "single-value" estimates. Provided we have a random sample from a normal population, we can make interval estimates of m or  $\sigma$  with a chosen degree of confidence.

 $\gamma$  - The confidence coefficient is simply the proportion of samples of size n for which intervals computed by the prescribed method may be expected to bracket m (or  $\sigma$ ). Such intervals are known as confidence intervals. As we would expect, large (size) samples tend to give narrower confidence intervals for the same level of confidence.

- 9. Statistical Tolerance Limits: For a given population, are limits within which we expect a stated proportion of the population to lie with respect to some measurable characteristics.
- 10. Engineering Tolerance Limits: Are specified outer limits of acceptability with respect to some characteristic usually prescribed by a design engineer.
- 11. Choice of Null and Alternative Hypotheses: Null hypothesis is considered to be the hypothesis under test as against a class of alternative hypotheses. The null hypothesis acts as a kind of "origin" or "base" (in the sense of "base line") from which the alternative hypotheses deviate in one way or another to greater and lesser degrees.

#### 12. Two Kind of Errors:

Error of the First Kind: If we reject the null hypothesis when it is true; e.g., announce a difference which really does not exist.

Error of the Second Kind: If we fail to reject a null hypothesis when it is false. Although we do not know in a given instance whether we have made an error of either kind, we can know the probability of making either type of error.

- 13. Significance Level and Operating Characteristics (OC) Curve of a Statistical Test:
  - $\alpha$  Level of Significance, is the risk of making an error of the first kind.  $\beta$  The risk of making an error of the second kind varies, as one would expect, with the magnitude of the real difference and is summarized by the Operating Characteristics (OC) curve of the test. Also, the risk  $\beta$  increases as the risk  $\alpha$  decreased "Only with large samples can we have our cake and eat it too" and then there is the cost of the test to worry about.
- 14. Choice of the Significance Level: The significance level  $\alpha$  of a statistical test should be chosen in the light of the attending circumstances, including cost. We are occasionally limited in the choice of significance level by the availability of necessary tables for some statistical tests. Two values of  $\alpha$ ,  $\alpha$  = .05 and  $\alpha$  = .01, have been most frequently used in research and development work and are given in tabulations of test statistics. We have adopted these "standards" for the purpose of this study.
- 15. Confidence Intervals for the Population Mean When Knowledge of the Variability Cannot be Assumed:

Problem: What is a two sided 100 (1 - Q)% confidence interval for the true mean m?

#### Procedure:

- 1. Choose  $\alpha$ .
- 2. Compute  $\overline{X}$  and S.
- 3. Look up t =  $t_{1-\alpha/2}$  for n 1 degrees of freedom (page 583, reference 28).

4. Compute:

$$X_{Upper} = X + t \qquad S/\sqrt{n}$$

$$X_{Lower} = X - t S / \sqrt{n}$$

Conclusion: i.e., we may assert that with 100 (1 -  $\alpha$  )% confidence that  $X_L < m < X_U$  or the interval from  $X_L$  to  $X_U$  is a 95% confidence interval for the lot mean; i.e., we may assert with 95% confidence that  $X_L < 1$  ot mean  $< X_U$ .

16. Number of Measurements Required to Establish the Mean With Prescribed Accuracy:

#### Procedure:

- 1. Choose d, the allowable margin of error, and  $\alpha$ , the risk that our estimate of n will be off by d or more.
- 2. Look up  $t_1 \alpha_{/2}$  for  $\nu$  degrees of freedom (page 583, reference 28).
- 3. Compute n or sample size:

$$n = \frac{t^2 s^2}{d}$$

Conclusion: We may conclude that if we now compute the mean  $\bar{X}$  of a random sample of size n from the population, we may have 100  $(1 - \alpha)$ % confidence that the interval  $(\bar{X} - d)$  to  $(\bar{X} + d)$  will include the population mean m.

- 17. Amines: Organic derivatives of ammonia; these may be primary, secondary, or tertiary, depending on the number of hydrogen atoms that have been replaced by organic radicals.
- 18. Radical: A group of atoms which occurs in the molecules of a number of compounds, and which remains unchanged through many chemical reactions. Example: Ethyl  $C_2H_5$  (valence 1)

- 19. Valence: An intiger representing the number of hydrogen or chlorine atoms which one atom of an element can hold in combination. Note: groups of atoms, i.e., radicals, also have valences.
- 20. Amino Acids: A compound that contains both an amine group and a carboxy group.
- 21. Carboxyl (or the compound name Carboxylic): A term for the -COOH-group, the radical characteristic of all organic acids.
- 22. Polyamide Resins: Resins made from polymerized vegetable oil acids.
- 23. Polymerized Oils: Vegetable and animal oils which have been heated and agitated by a current of air or oxygen. They are partially oxidized, deodorized, and polymerized by the treatment and are increased in density, viscosity, and drying power.
- 24. Polymers: A molecule made up of hundreds, thousands, and even tens of thousands of repeating units called monomers.
- 25. Monomers: Are usually simple, reactive organic molecules which react with one another to link together over and over again and form giant molecules, the polymers, which often have molecular weights running into the millions.
- 26. Polymerization: A union of monomers.
- 27. Condensation Products: Some polymers are formed by condensation reactions which, in most instances, involve the elimination of a simple molecule such as water or ammonia.
- 28. Oxidation: In a broad sense, oxidation is the increase in positive valence or decrease in negative valence of any element in a substance.

  On the basis of the electron theory, oxidation is a process in which an element loses electrons. In a narrow sense, oxidation means the

- chemical addition of oxygen to a substance.
- 29. Exothermic Reactions: Chemicals combining to form stable compounds and which give off energy in the process.
- 30. Exothermic: Refers to a process which is accompanied by evolution of heat.
- 31. Epoxy: A prefix in organic nomenclature denoting an oxygen atom joined to each of two atoms which are already united in some other way, as -C-O-C.
- 32. "Epon" Resins: Trademark for a series of condensation products of epichlorohydrin and bisphenol A, having excellent adhesion, strength, chemical resistance and electrical properties when formulated into protective coatings, adhesives, and structural plastics.
- 33. Epoxy Resins: Thermosetting resins based on the reactivity of the epoxide group -C-C-
- 34. Epoxidation: The reaction of oxygen with an olifin (a class of unsaturated, aliphatic hydrocarbons of the teneral form  $C_n$   $H_{2n}$  and named after the corresponding paraffins by adding "ene" or ylene" to the stene: as ethylene, propylene, butenes, etc.) to form an epoxy compound.
- 35.  $F_X(x)$  = distribution function of the random variable X defined for any real number x.
- 36.  $f_{\chi}(x)$  = probability density function:  $f_{\chi}(x) = \frac{d}{dx} F_{\chi}(x)$
- 37. E[X] = the expectation, or mean of X, when it exists is defined by:  $E[X] = \int_{-\infty}^{\infty} x f_{X}(x) dx$

## APPENDIX C

# YATES' METHOD FOR OBTAINING ESTIMATES OF MAIN EFFECTS AND INTERACTION FOR TWO-LEVEL FACTORIALS

C	YATES METHOD FOR OBTAINING ESTIMATES OF MAIN EFFECTS AND
C	INTERACTION FOR TWO-LEVEL FACTORIALS
	DIMENSION TAB(32.5) . TEMP(32)
V	READ (READER : 500) N : ALPH : TA
500	FORMAT(15.5X.2F10.0)
h dd	ANEN
	Ngasaan
	RN#NR
James Start of Ballahaman	READ (READER . 505) ( (TAB(I.J) .J#1.3) .I#1.NR)
505	FORMAT (A5.5X.2F10.0)
C	PUT RESPONSE IN SAVE AREA
	DO 1 I#1.NR
1	TEMP(I)#TAB(I.3)
C	PERFORM N**2 ADDITIONS AND SUBTRACTIONS TO OBTAIN GT.GA.ETC.
С	FINAL VALUES OF G ARE IN COLUMN JJ OF TAB
	NRHANR/2
	JEB
	JURA
······································	DO 4 K#1.0N
-	IE\$2
	10%1
	D0 2 I#1 • NRH
·· • • • -	TAB([:JJ)#TAB([E:J)+TAB([O:J)
	TAB(I+NRH.JJ)#TAB(IE.J)-TAB(IO.J)
	15415+5
2	10%10+5
	IDUMAJ
	し掛しし

· · · · · · · · · · · · · · · · · · ·	
4	JJ#IDUM -3
	JJ#J
С	DIVIDE VALUES OF G BY 2**(N-1)
	DO 6 I#1+NR
6	TAB(I+5)#TAB(I+JJ)/(2.**(N-1))
С	CALCULATE S2 WHICH IS THE SUM OF THE SQUARES OF G S
	S2#0•
	DO 10 I#1•NR
	IF(TAB(I+2)-3.)10.8.8
8	S2#TAB(I+JJ)*TAB(I+JJ)+S2
10	CONTINUE
С	CALCULATE NU AND W
	UN#RN-0.5*(AN*AN+AN+2.)
	S2#S2/(RN*UN)
	W#TA*SQRT(RN*S2)
	DO 11 I#1+NR
11	WRITE(PRINTER+510)TAB(I+1)+TAB(I+2)+TEMP(I)+TAB(I+JJ)+TAB(I+5)
	FORMAT(10X+A5+5X+E14+2+3E20+8)
	FEED(3)
	WRITE (PRINTER • 515) S2 • W
E16	FORMAT(10X.3HS2#F15.8.10X.2HW#F10.5.//)
C 213	IF ABS(G(I)) IS GREATER THAN W THEN PRINT G(I)
•	DO 14 I#1•NR
	IF(ABS(TAB(I+JJ))-W)14+14+12
	WRITE (PRINTER+520) TAB(I+JJ)
520	FORMAT(E20.8)
14	CONTINUE
	END

#### APPENDIX D

# DISTRIBUTION OF THE NEAREST NEIGHBOR IN A POISSON DISTRIBUTION OF FIBERS IN SPACE

Reference 40 was used for the following analysis. If particles or fibers are distributed in a 3-dimensional space in accordance with a Poisson process at a mean rate or intensity  $\mathcal V$  per unit volume, then

$$p (K; \nu V) = e^{-\nu V} (\nu V)^{K}$$

$$K!$$
(D-1)

considers the probability of finding exactly K particles in a fixed volume V. In particular, the probability of no particles in a volume V is

$$p(0, VV) = e^{-VV}$$
 (D-2)

and the probability of one or more particles is therefore

$$1 - e^{-\nu v}$$
 (D-3)

From Appendix B, definition 35,

$$F_{X}(x) = P \left[ X \le x \right]$$
 (D-4)

and from definition 36

$$f_{\chi}(x) = \frac{d}{dx} F_{\chi}(x)$$
 (D-5)

If  $\delta$  is the distance between an individual fiber and its nearest neighbor, then

$$p (0, \nu \frac{4}{3} \pi \delta^3) = e^{-\nu \frac{4}{3} \pi \delta^3}$$
 (D-6)

or

$$f_{\mathcal{S}}(x) = \frac{d}{dx} \in -V^{\frac{4}{3}} \Pi X^{3}$$

$$= 4 \Pi V X^{2} \exp \left\{ -\frac{4}{3} \Pi V X^{3} \right\}$$
(D-7)

To find the mean distance, E[S], between a fiber and its nearest neighbor, definition 37 of Appendix B is used:

$$E\left[\delta\right] = \int_{0}^{\infty} x f_{(\delta)}(x) dx \qquad (D-8)$$

hence

$$E\left[\delta\right] = \left(\frac{3}{4\pi}\right)^{1/3} \left(\frac{4}{3}\right) \mathcal{V}^{-1/3}$$

or

$$E\left[\delta\right] = 0.554 \quad \mathcal{V}^{-1}/_{3} \tag{D-9}$$

where  $\mathcal V$  is equal to the number of fibers per unit volume. The fiber volume ratio

$$V_{R} = \frac{\eta^{V}f}{V_{T}}$$
 (D-10)

Substituting equation (D-10) into (D-9) for  ${\cal V}$  , the following expression, for  $\delta$  , is obtained:

$$\int = \frac{0.554}{\sqrt[3]{V_{R/V_F}}}$$
(D-11)

where  $V_R$  is the fiber volume ratio and  $V_f$  is the volume of 1 fiber. Once  $\delta$  is determined, the average thermal conductivity is found in a similar procedure outlined in Part II, Section A.

0r

$$K_{B} = \frac{K_{f} K_{m}}{\left(\frac{\delta}{L}\right) K_{f} + \left(I - \frac{\delta}{L}\right) K_{m}}$$
(D-12)

where L is the length of the model. Note that if  $\delta$  is equal to zero,  $K_B$  is equal to  $K_f$ . Results from this theory are presented in Tables XIV through XVII.

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randomly distributed in low conductive matri	ces, is inve	stigated e	experimentally as well
as theoretically.			
Experimental results are present			
five factors at two levels. The factors are	the length	of the fib	ore, the cross section
of the fibre, the thermal conductivity of the	e fibre, the	thermal c	conductivity of the
matrix, and the fibre volume ratio.	C1 C		sidemed for an entimum
It is shown that the most meaning			
design of the type of composites in question	are, in the	fibre cre	se section
ductivity of the matrix, the fibre volume ra Earlier theoretical efforts cons	icio, and the	homotical	models which are
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nighty idealized, e.g. square lattices, late	s centered co	t of a mat	riv filled with fibres
Bruggeman). The model used in this investig	gation is tha	t or a mac	ibod fibro volume
ellipsoidal in shape and randomly distribute	ea, according	conductiv	vity of this composite
ratios. It is a stochastic model. An expre	ession or the	ch speed s	computer Theoretical
is derived and results are obtained with the	aid of a Ni	gn-speed C	Also it shows that
prediction of the stochastic model checks wi	in experimen	uala.	not covered by
additional factors are involved in describing	ig composites	wnich are	bution
earlier theories. These factors are fibre	geometry and	res distri	mal conductivity
Moreover, a specific specimen for	ormulation gi	ves a thei	f that of the steel
similar to that of stainless steels but a de	ensity only a	quarter o	or that or the steel.

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